Chemical & Process Engineering

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Topics of the Month

More chemicals from coal

'COAL is our most wasted asset.' This is not a recent observation of some well-known industrialist. It was said a century ago by Stanley Jevons, first Professor of Political Economy in the University of Manchester. Paying tribute to him as the ' father of fuel efficiency,' the Minister of Fuel, at the annual luncheon of the Institute of Fuel, recalled the grim warnings uttered by Jevons concerning the dangers into which inefficient use of fuel would lead us.

Jevons has long since passed away and the chimneys have gone on relentlessly pouring out their black smoke. Today, industry is only too acutely conscious of the dwindling of our coal resources. The chemist also looks with concern at our limited reserves. For, to him, coal is not merely a source of energy for industrial operations. It is a precious heritage of hydrocarbons of which he is spared only the crumbs from the plate of the carbonising industries. Elsewhere in this issue this problem is touched upon in two articles. One, dealing with the recovery of by-products from coke-oven gases, emphasises that large percentages of valuable hydrocarbons are lost in the high-temperature process, and hints at a possible means of improving the yield. The other describes an ambitious American venture to produce aromatic chemicals *directly* from coal by an improved process of hydrogenation. This may open up a completely new field of aromatic chemicals, many of them becoming available in large commercial quantities for the first time, and ultimately much more cheaply than at present.

The importance of the process is that for the first time coal is treated directly as a chemical raw material. If it proves successful, the chemical industry will no longer be dependent upon the coking industry's by-products for its coal-derived raw materials. The situation was neatly summed up by an official of Carbide & Carbon Chemicals Co. Inc., the operators of the new plant, who said ' The steel men are interested primarily in producing metallurgical coke and any by-products they obtain are for sale, but the tail can't wag the dog and the chemical industry either has to produce its own primary source of essential raw materials or slow down its pace to that of the supply.'

I.Chem.E. entrance standards

AS briefly reported in our last issue (p. 155), the president of the Institution of Chemical Engineers, Mr. Stanley Robson, had some important things to say about chemical engineering training at the Institution's annual dinner. The most significant comment was on conditions of entrance to the Institution. The Council is now considering proposals for a completely revised examination. If these are accepted the result will be an examination certainly no less

rigorous than the present one. If anything, it will be more rigorous. Certainly no other interpretation can be given to Mr. Robson's advice that those contemplating taking the present examination should do so quickly. These proposals are in accord with the Institution's determination to maintain the highest standards in the profession. With this in mind it was all the more encouraging to learn that between the two Coronation years (1937 and 1953) membership of the Institution has risen from 900 to 2,900. Mr. Robson rejoiced particularly in the strength of the junior membership, which probably exceeds, at least in quality, that of comparable institutions and shows plainly that chemical engineering is indeed a profession of the future.

What types of chemical engineer are needed?

THE engineer-chemist combination is a European tradition which has produced some of the great achievements of the chemical industry. This combination is still used by some powerful companies for the very good reason that it continues to serve them well. Chemical engineers are naturally impatient with this attitude. Indeed, in the O.E.E.C. report, *Chemical Apparatus in the U.S.A.*, which was published last year, the following criticism appears: 'The old European tradition of relying entirely on the combination of the industrial chemist and the mechanical engineer seems outmoded and has now become a vested interest tending to stifle chemical engineering progress in Europe.'

The question 'Do we want the chemical engineer and if so what do we want of him?' is revived by a thoughtful article in the April issue of the *I.C.I. Magazine* by J. E. Braham, the engineering controller. I.C.I. is a company which has in the past relied very much on the engineer-chemist combination and, indeed, still uses it. Mr. Braham, nevertheless, concedes the wide acceptance that the chemical engineer can be of real use in industry. He goes on to ask and discuss the questions 'What types of chemical engineer does industry need, and can the present facilities for training, both from the theoretical and the practical point of view, produce what is required in quantity and quality?'

A recent survey in I.C.I. revealed the widespread opinion that the technical training of a chemical engineer would best be based on that of a mechanical engineer, extending usually over three years, followed by chemical engineering training proper on a post-graduate basis for one or preferably two years. However, Mr. Braham points out that this preference does not mean there is no place for the chemical engineer with a basic training in chemistry or physics. He continues: 'The chemical engineer trained only on a specialised chemical engineering basis cannot, of course, by virtue of that training, be either a fully qualified chemist or physicist or mechanical engineer. His place in industry is correspondingly of a more specialised nature, which is not to say, however, that there are only a few activities for which he is suitable. More so than the chemical engineer who was originally a chemist or originally a mechanical engineer, he will require a correspondingly greater measure of support from the straight mechanical engineer or the straight chemist.

'The volume of present-day knowledge is already so great that none apart from the exceptionally talented can possibly expect to achieve a comprehensive grasp of all that is required of engineers in the process industries. Chemical engineers, however widely they are used, must still be used in conjunction with chemists, physicists, mechanical, electrical and civil engineers, instrument technologists and materials specialists.

'The chemical engineer should be a man who can operate over a wide field and whose most frequent role will be to act as an effective link between the chemist and the mechanical engineer. In general, he will be employed as an additional member of a technical team, so broadening and strengthening the whole structure that problems associated with the rapid advance in scientific knowledge, and particularly their practical application, can be dealt with effectively.'

This definition of the place of the chemical engineer in industry will not be accepted by all, but, coming from so important a source, it must command serious attention. We would welcome our readers' views on it.

Fresh thinking in atomic energy

A NEW phase in the development of atomic energy in Britain is in prospect now that the Government has decided to transfer this responsibility from the Ministry of Supply to a non-departmental body. This decision, announced by Sir Winston Churchill in Parliament recently, appears to have been taken as a result of the many criticisms of the present arrangements which have been voiced through the years. While it was reasonable in the early days to place the development of atomic energy under the Ministry of Supply, this has come to be seen more and more as an outmoded administrative expedient as the programme has grown and increasing sums of money have been poured into new installations and new projects. Clearly, atomic energy, with all its military and industrial implications, is now too big a project to handle as an appendage of a Government department. A separate organisation on the lines of the Atomic Energy Commission of the U.S. is needed. Three eminent men, Lord Waverley (formerly Sir John Anderson), Sir Wallace Akers (just retired as research director of I.C.I. and Director of Atomic Energy Research, 1941-46) and Sir John Woods (a director of English Electric), have been named as members of the committee which is to plan the new set-up. They will have a difficult job to devise an organisation which will combine the security of a Government department with the necessary vigorous and adventurous outlook needed to thrust the programme forward.

Meanwhile, in speculating on the nature of the new atomic energy organisation, we should not forget the achievements of the Ministry of Supply. These were concisely stated by the Minister of Supply in Parliament last month when he was asked to explain the present ramifications of the atomic energy programme. There are now six main establishments. That at Harwell, in Berkshire, is responsible for fundamental research into nuclear physics and atomic energy, and provides basic scientific information to the other establishments.

The radiochemical centre at Amersham, in Buckinghamshire, is a dependency of Harwell. It is concerned with preparing radioactive substances such as radium, radon, and radioactive isotopes produced in the atomic piles, and these are used for medical, scientific and industrial purposes.

The production of fissile material is directed from headquarters at Risley, near Warrington, where the design and planning offices are situated. There are factories at Springfields, near Preston, producing pure uranium from uranium concentrates; at Windscale, in Cumberland, where plutonium is produced from uranium by means of atomic piles; and at Capenhurst, in Cheshire, where there is a gaseous

Loyal Greetings to

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On the occasion of her Coronation, June 2, 1953

diffusion plant for separating uranium isotope U235 from the more abundant isotope U238. This provides fissile material which can be used as an alternative to plutonium.

Research work on atomic weapons is carried on at Aldermaston, Berks, and its out-stations at Woolwich, London, and Fort Halstead, Kent.

Uranium recovery

AS is well known, the American Atomic Energy Commission makes far greater use of private industry than its British equivalent. An example is the new laboratory for the development and improvement of processes for the recovery of uranium from its ores at Winchester, Massachusetts. This is being operated for the Commission by the American Cyanamid Co. Originally the laboratory was located at the Watertown, Massachusetts, Arsenal where it was operated for the A.E.C. by the Massachusetts Institute of Technology, under the direction of Dr. A. M. Gaudin, Professor of Mineral Engineering at the Institute. As the operations grew in size, the M.I.T. decided it could no longer do work which rightfully should be done by industry. In 1950, therefore, American Cyanamid was awarded the contract, continued work at Watertown until December last, and then transferred to the new laboratory at Winchester.

The laboratory is well equipped for studying all phases of mineral dressing, and one of its functions will be to undertake the vitally important task of developing methods for effective and economical recovery of uranium from low-grade ores and other low-grade uranium-bearing materials.

Ore samples from uranium fields throughout the world are sent to the laboratory in Winchester by the Atomic Energy Commission. Sometimes very small quantities are involved and sometimes several wagonloads.

Basically, the operations of the laboratory are those of a commercial metallurgical processing mill. The experience gained is passed to producing units, which put the processes into effect at installations on or near the sites of uranium-bearing ores. The Winchester laboratory, in other words, starts with the ore and carries it through to the end product, which is a uranium concentrate of acceptable grade and composition. In addition, field parties of laboratory engineers are frequently sent to plants in America and in other countries so that they may give technical advice concerning processes which have been developed.

An example of this work was the recent erection of a small producing unit and analytical laboratory at Grand Junction, Colorado, which is staffed with Winchester personnel. If the unit proves satisfactory, the recovery of uranium from certain types of ores will be improved and costs cut considerably.

Plant exporters' problems

SHORTAGE of raw materials and delay in obtaining components have been two big obstacles faced by chemical plant exporters in the past year. Because of them, makers have not always been able to keep delivery dates and have thus lost goodwill which will not be regained easily. These difficulties are publicised in the 1952 report of the British Chemical Plant Manufacturers' Association, which was issued just after the recent annual meeting.

Other big difficulties are the limitations imposed by import licensing and exchange control and the steady growth of foreign competition, especially from West Germany. There is widespread dissatisfaction with the facilities afforded by the Export Credits Guarantee Department, which do not enable members to match the specially advantageous terms of payment offered by many of their foreign competitors, and the Association would welcome a substantial improvement in these facilities.

Foreign competitors are further assisted by the export incentive devices such as dollar retention and tax remission schemes made available by their governments. These incentives have been fully discussed by the B.C.P.M.A. Council with Sir Norman Kipping, director-general of the Federation of British Industries. The Association supports the Federation's view that the United Kingdom should press through international organisations for the complete abandonment of such incentives rather than invite further competition in their use by itself adopting them; like the Federation, it believes that increased production for export can best be stimulated by the removal of fundamental deterrents such as the heavy burden of corporate and personal taxation.

Protein extraction mill

PLANTS, although excellent sources of carbohydrates, fats and vitamins, are generally poor sources of protein. However, there are two outstanding exceptions: the seeds of many legumes (beans and peas) and the young leaves of many plants.

According to N. W. Pirie, who contributed a paper with the arresting title of 'The efficient use of sunlight for food production' to a recent Society of Chemical Industry symposium 'Food and the Future,' young, vigorously growing leaves often contain 30 to 40% of protein and little fibre. If suitably selected they would be valuable food for people and research should be directed towards establishing the conditions under which leaves of this very high quality could be produced regularly. They could be used as food either directly or after sufficient treatment to remove unattractive flavours. The lower-quality leaves could be fed to animals, but it would be more efficient to try to process

them so that they could be used also as human food. Their defect is that, as the protein content falls the fibre content in general goes up, and it is this that makes the leaf unsuitable for use as food by non-ruminants. The solution lies in separating proteins and digestible fats and carbohydrates from the fibre by mechanical or chemical means.

Even primitive peoples carry out similar separations of valuable foodstuffs from inedible surrounding material; as examples, the processes of shelling peas and threshing and milling wheat may be mentioned. Except for a change in scale, the protein with the leaf is essentially similar; here we are concerned with the separation of the cell wall from the cell contents, whereas the other processes separate organised cellular structures from one another. The fluid cell contents, after liberation, bring out with them most of the leaf protein, starch and fat as well as sugars and other components of low molecular weight. The separation of protein has been carried out on a laboratory scale with varying degrees of efficiency for 180 years, and several attempts have been made during the last quarter century to do it commercially. So far these have not been an unqualified success. This is partly because people have tried to use machinery developed for one purpose for a quite different purpose. Thus the standard three-roll sugar-cane mill, which is the result of 500 years of trial and error, and the screw oil expeller can both be made to work. But they work inefficiently, mainly because they apply pressure without sufficient rubbing, and what rubbing there is goes on extravagantly on a tightly packed mass. The old manufacturers of woad had a better plan when they macerated fresh leaves on an open bed with a corrugated roller and then pressed out the juice. After much experimenting, the same principle has now been adopted, but in a modified form to make the mill continuous in action.

The mill operates as follows: A number of beater arms revolve inside a drum with about ½ in. clearance from the wall; they are of two types—one propeller-shaped to move the charge along the drum and the other U-shaped so that it simply beats. By suitably arranging the ratio of these two and the speed of rotation, the mill is adaptable to crops of differing texture, so that succulent ones go through very much more quickly than fibrous ones and all come out adequately ground. For the expenditure of 20 h.p. as much as 6 tons of very succulent material an hour can be ground, and the rate only falls below 1 ton/hr. on material that is so dry that it is hardly worth processing. So far, a machine that is so satisfactory for pressing the juice out from the ground mass has not been designed. Somewhat fuller particulars of these machines have been given in our associated journal, *World Crops*, 1952, 4, 374.

Acid conservation and recovery

THE Coatings committee of the British Iron and Steel Research Association has been giving a lot of attention to the conservation of sulphuric acid in the pickling process and its recovery from waste liquor. According to the 1952 annual report of the Association, these researches have led to the conclusion that the best way of recovering unused acid from waste liquor is by evaporating the liquor to high acid strength, whereupon ferrous sulphate separates out as the monohydrate. Whether this is done by the submerged combustion method or by the use of a heat exchanger will depend, for a particular works, on the fuels available and the use for which the ferrous sulphate is intended. Whereas the submerged combustion method leads to the formation of crystal aggregates, the heat exchanger method yields very small single crystals. The best conditions for producing large single crystals ot ferrous sulphate monohydrate are the subject of further investigations.

Another subject to which attention has been given is the influence of bath composition on the rate of pickling. One result of these experiments has been to indicate that an increase of 10% over present normal acid strength may permit an increase of 50% in the pickling rate.

More and more industries are becoming interested in the activities of B.I.S.R.A., whose research programme has been steadily expanding over the years to embrace ever-widening fields of processes and operations. Besides its main research into the processes of ironmaking, steelmaking, metalworking, plant engineering and metallurgy, the Association helps to sponsor, in various ways, researches on subjects such as corrosion, deep drawing and pressing, and cutlery manufacture, which lie on the fringe of its own activities.

Whereas in 1945 the industrial income of the Association was about £64,000, earning a Government grant of £30,000, in 1952 it was over £312,000, which earned the maximum Government grant of £150,000. During this eight years the staff has grown from a small nucleus to the present total of nearly 400.

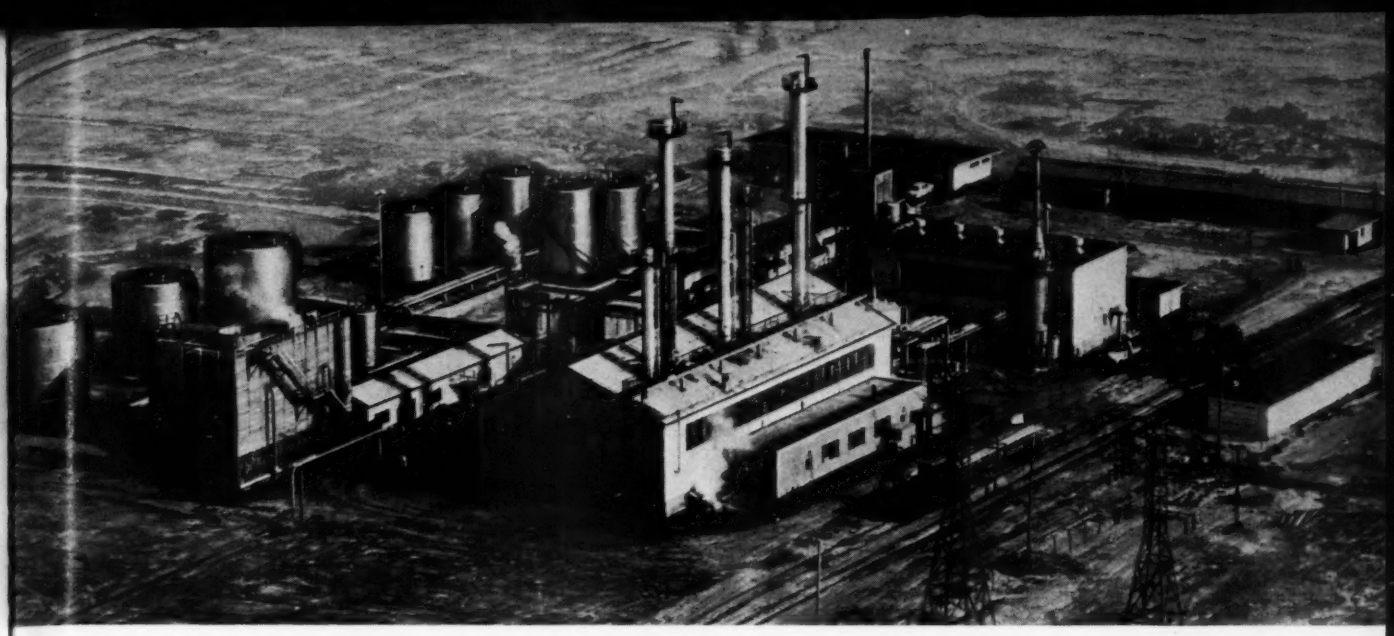
Fighting corrosion

AN essential part of B.I.S.R.A. is the Chemistry Department, which shares with the Physics Department the prime responsibility of supporting and giving guidance to the work of the five main divisions. The Physical Section of the Chemistry Department carries out theoretical research on the thermodynamics of various elements in iron and steel, on vacuum fusion analysis, the constitution of slags, and other subjects. The Refractories Section takes part in collaborative work with the British Ceramic Research Association on the design of furnaces and the testing of refractories. Then there is the work of the very important Corrosion Section, which includes experiments with protective metallic coatings and paints and the carrying out of widespread tests on metals left exposed to atmosphere over a certain period. We mentioned these experiments in our February issue.

The setting properties of the acetate type of cementiferous paint have been studied and the difficulties mainly overcome; there are indications that mixed acetate-phosphate paints will prove superior to the paints in either medium alone.

It has been found that the film formed on iron by the anodic discharge of hydroxyl ions is composed of material having the cubic structure of either magnetite or γ-ferric oxide; consequently it is indistinguishable from the air-formed film. This film may be produced when freshly abraded iron is immersed in decinormal sodium hydroxide solution containing dissolved oxygen, or in decinormal solutions of the sodium salts of weak acids, such as phosphoric, boric or carbonic acids, which are readily hydrolysed.

Another discovery of the Corrosion Section, in work on rust-resisting steel, is that there is a rectilinear relation between the amount of oxygen added to dilute sulphuric acid in which the steel is immersed and the reciprocal of the time needed for passivity. This indicates that the growth of the protective film follows a logarithmic law. Analogous work carried out using potassium dichromate in place of oxygen gives similar but less regular results.



SOLVENTS PRODUCED IN CANADA

Engineering Details of Shell's New Petrochemical Plant

First of its kind in Canada, the Shell Oil Co. of Canada's new petroleum chemical plant at Montreal East was officially opened on April 14, 1953. The plant will produce 1½ million gal. of isopropyl alcohol and 1 million gal. of acetone annually. These chemicals, the raw material for which will be drawn from nearby refinery units, will be used in such varied industries as synthetic textiles, medicine and paint manufacture. Process design for the new plant, based on 20 years' experience of petrochemical processing in England and America, was in the hands of the company's own engineers. In building the plant, special regard has been paid to Montreal's extreme climate. Interior plant temperature, for example, will remain at 40° F. even when outside temperatures fall to 20° below zero. Specially heated storage tanks will ensure maintenance of even storage temperatures, while an elaborate air-conditioning system protects personnel from any noxious fumes which may arise in the course of refinery processes.

CANADA'S first production of isopropyl alcohol on a commercial scale is now under way at Shell Oil Co. of Canada's new petrochemical plant near Montreal. This plant, the first petrochemical plant to be owned and operated by an oil company in Canada, is situated adjacent to Shell's Montreal East refinery. Ground was broken for the project in April 1952 and less than one year later the plant was placed on stream. As well as isopropyl alcohol, acetone is also being produced, the combined production approximating 20 million lb. p.a.

Feed to the plant consists of a liquid mixture of propane and propylene, produced from Shell's adjacent catalytic and thermal crackers. The first step in the process is the reaction of the propylene with sulphuric acid. As this step is strongly exothermic, coolers are provided for removing excess heat. The ester thus formed is subsequently hydrolysed by dilution and mixing with water, forming crude isopropyl alcohol in a mixture with dilute sulphuric acid. The unreacted propane is caustic-scrubbed, dried and subsequently sold as L.P.G. The separation of the crude isopropyl alcohol from the dilute acid is accomplished by stripping, using steam as the stripping medium.

Dilute sulphuric acid leaves the bottom of the stripper and is subsequently reconcentrated in a Chemico drum concentrator and returned to the system.

The next step in the process, which involves distillation, removes isopropyl ether formed as a reaction by-product, and reduces the water content of the crude isopropyl alcohol to about 13% by weight, approaching the constant boiling point mixture.

For the production of acetone, isopropyl alcohol is catalytically dehydrogenated in a Petrochem furnace. The crude acetone, formed by the removal of hydrogen from the isopropyl alcohol molecule, is separated from the reaction by-products and purified to 99.5% by weight purity or, better, by means of a scrubbing step followed by distillation.

For the production of finished isopropyl alcohol, two steps are required. The first of these consists of an azeotropic distillation in which the water content of the alcohol-water mixture is reduced to approximately 1%. Finished isopropyl alcohol is then produced by final distillation, which serves to remove materials boiling below and above the pure alcohol boiling point.

Instrumentation and engineering

The entire operation is highly instrumented in the interests of precise operating control and to reduce manpower requirements. Reaction, hydrolysis, stripping and all distillation equipment is housed in one large building. The acid concentration equipment occupies another building, and transfer and cooling tower pumps a third. An adjacent tank farm provides storage for rundown, intermediate and finished products. Shell turbo-grids are used in many distillation applications.

In an enclosed process of this nature, the collection of pump drips and of the blow-downs from gauge glasses and sample pots is of importance both from the standpoint of minimising vapour evolution and also for reducing losses. Hydrocarbon and acid drips and blow-downs in all buildings are gathered by means of enclosed collection headers connected to sumps equipped with level-controlled pump-outs to storage tanks.

Winterising of a process designed to operate in a climate of severe temperature

fluctuations and at the same time requiring the use of many high-freezing-point materials presented unique problems. The Montreal plant is designed to operate in weather temperatures as low as –20°F. The plant is divided into two basic areas; the hazardous area where some inflammable vapours may exist and the non-hazardous area in which the fired equipment is located. All process equipment in these areas, with the exception of a Petrochem furnace, is entirely enclosed within buildings.

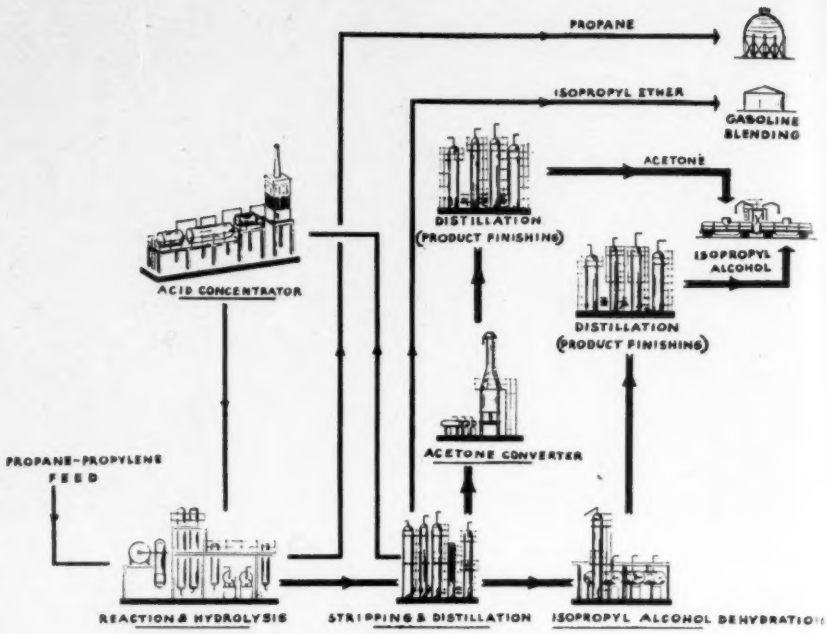
Plant details

The process building is approximately 65-ft. wide by 140 ft. long by 28 ft. high. It has a rigid steel frame, pitched roof, and trafford tile roof and siding above a brick wainscote. Projection-type unit heaters near the roof heat the building to an average temperature of 40°F. when the outside temperature is –20°F. The heating is thermostatically controlled. Ventilating units at the ends of the building supply a total of 65,000 cu.ft./min. of outside air the year round, tempered to 40°F. in the winter. The air is exhausted near the floor by 12 fans, six on each of the two sides of the building, providing a complete air turnover every 6 min.

The control building is 20 ft. wide by 118 ft. long. It is attached to the process building and divided into three sections: a central control room for both the reaction and distillation process adjoining the process building and connected with sufficient windows to allow vision of the process equipment from inside the control room; a control laboratory on one end and a utility room on the other. The utility room houses the instrument air compressor and drier, the ventilating equipment and water heater. This entire section is ventilated with filtered outside air distributed to each room. The total air-quantity is 3,000 cu.ft./min. in the winter and 600 cu.ft./min. in the summer. The building is pressurised all the year round.

The acid recovery plant located in the non-hazardous area houses a two-stage Chemico unit, affiliated pumping, acid blending and cooling equipment and a control room. The acid mist precipitator and substation are adjacent to the main building. The precipitator is designed to remove 93% of the sulphuric acid mist entrained in the effluent steam and furnace stack gases. The elimination of a public nuisance rather than economic considerations is the justification for this installation. The concentrator building is 40 ft. wide by 78 ft. long by 30 ft. high and it has a pre-cast reinforced-concrete frame, pre-cast concrete roof and trafford tile siding above a brick wainscote. The heating and ventilation system is similar to that of the process building.

A pumphouse is situated adjacent to the tank farm area and houses all product transfer pumps, some of the process feed pumps and the cooling water circulating pumps. Thermostat-controlled unit heaters maintain a 40°F. temperature during minimum winter temperatures.



Flowsheet of processes used at the new solvents plant

Outdoor installations of instruments are protected from the weather by sheet-metal enclosures heated by internal steam coils. All instrument boxes have adequate access doors or panels for operation and maintenance.

Winterising of cooling towers presents unique problems in a climate such as Montreal's. A two-cell induced-draught tower is used. The fans operate at two different speeds to meet various winter conditions and also are equipped with a reversing switch to enable the towers to be de-iced by means of a downward flow of air. Removable wooden panels are provided for covering the air louvres in the winter.

Storage of sulphuric acid in –20°F. presents still another problem: freezing of acid with insufficient heat or accelerated corrosion if overheated. All acid tanks are insulated and heated with external steam coils located between tank pier supports with sufficient gap between coils and tank bottom to prevent hot spots. Tanks are heated and insulated to maintain acid at 60°F. during –20°F. weather.

Safety

Much attention has been given to safety features. The separately pressurised control room, as well as the rapid air turnover in the process and concentrator buildings, minimise the exposure of operators to fumes. The portion of the reaction equipment which circulates acid mixtures at high pressures is separated from the rest of the building by a high bulkhead. Windows are installed for observation of the equipment and operating and drop-out valves extend through the bulkhead. Many of the pump glands and process valves are provided with shields to contain leaks. An elaborate alarm system has been installed to call attention to faulty operating conditions.

Corrosion

In a plant handling dilute sulphuric acid at elevated temperatures and pressures, the battle against corrosion is an unceasing one. The Shell organisation's many years of experience in designing and operating petrochemical plants was called upon for the solution of many tricky corrosion problems. Such materials as high-silicon iron, *Monel*, carbon, *Chlorimet*, *Lebanon* 34, *Colomonoy*, *Hastelloy*, tantalum, *Teflon*, lead and copper have been used. Where concentrated acid is stored in unlined steel tanks, advantage was taken of a process termed ' chilled welding ' to reduce groove corrosion commonly experienced adjacent to welds. This type of corrosion is usually considered to be due to spheroidising of the steel as a result of the heat generated during welding. ' Chilled welding ' consisted of cooling the opposite side of the tank from which the welder is working with a spray of water, while using a low-hydrogen mild-steel rod. By this means the length of time at which the steel was held in the critical temperature range was reduced. The first seal weld, which must be applied without water cooling, is chipped out before being covered by subsequent passes on that side of the tank. Tests on sample specimens have indicated that groove-type corrosion can be appreciably reduced by this procedure.

(*Concluded on page 170*)

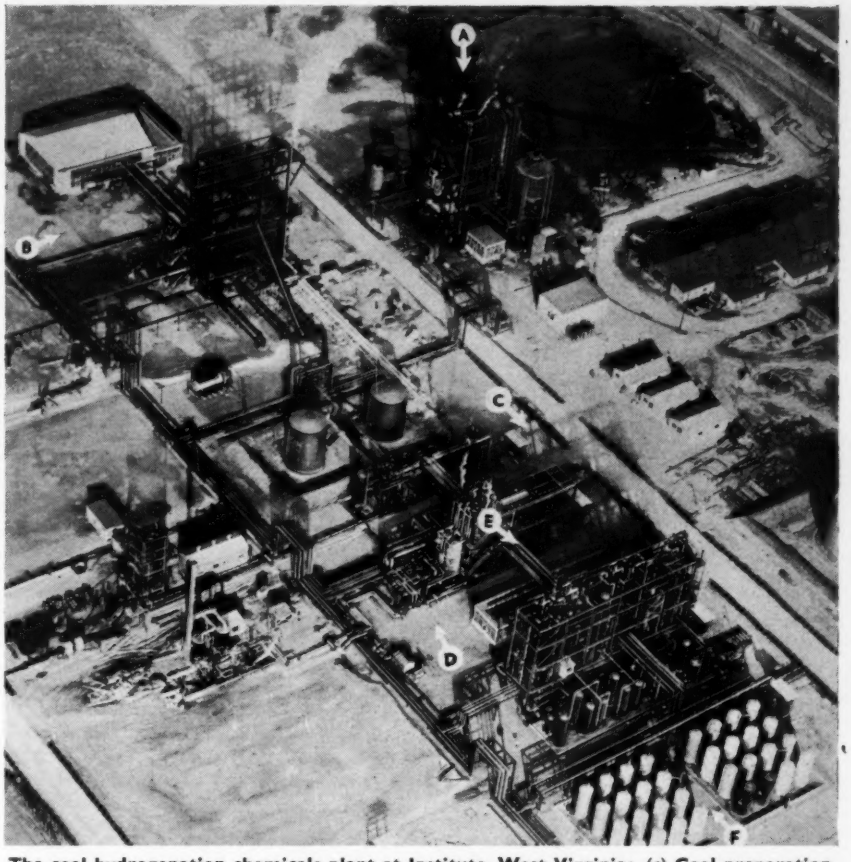
Coal Hydrogenation Chemicals Plant

What is claimed to be the first plant of its kind in the world has been put into operation at Institute, West Virginia, by Carbide & Carbon Chemicals Co. It is designed to process 300 tons/day of coal to produce a large range of basic aromatic chemicals. Although coal hydrogenation is not new, this is the first time it has been used to produce chemicals instead of fuels. Here is a description of the plant, its operation and products.

THE mounting demand for aromatic chemicals has led to increasing realisation that dependence on by-product coke ovens has considerable drawbacks. Except for benzole and toluol—both somewhat painfully twisted out of certain limited fractions of petroleum—and some cresylic acids, the only sources of the basic raw materials for the entire aromatic chemical industry are coal tar and other by-products of the iron and steel industries' coke ovens. New end uses are increasing the demand for some aromatic chemicals by a much greater percentage than is shown in the trend of increased by-product production from coke ovens.

A need has long been felt for a process for producing chemicals directly from coal as *primary* products. Carbide & Carbon Chemicals Co. believe that coal hydrogenation is the answer, and that the direct production of chemicals from coal by this process, carried out on a large scale, is an attractive proposition. Their $11-million experimental plant, spread over a 20-acre site at Institute, West Virginia, is a token of the strength of their conviction. The plant, which was opened in the spring of 1952, was originally designed to process 300 tons/day of coal, but it is hoped to step this up eventually to 500 to 600 tons/day. From the operating data obtained it is planned to build a much larger plant in the future. This will probably be composed of units each of which will have a coal-processing capacity of 3,000 tons/day.

Not only is this the first time that a *chemical* company has used a raw material for the sole purpose of producing chemicals, but it marks the first major development in coal hydrogenation since the Bergius process was first made use of in Germany. Before going into further details of this new project, a brief history of the coal hydrogenation process to date might prove of interest.



The coal hydrogenation chemicals plant at Institute, West Virginia: (*a*) Coal preparation structure; (*b*) high-pressure building; (*c and d*) chemicals separation unit; (*e*) product receivers and product storage tanks

Development of coal hydrogenation

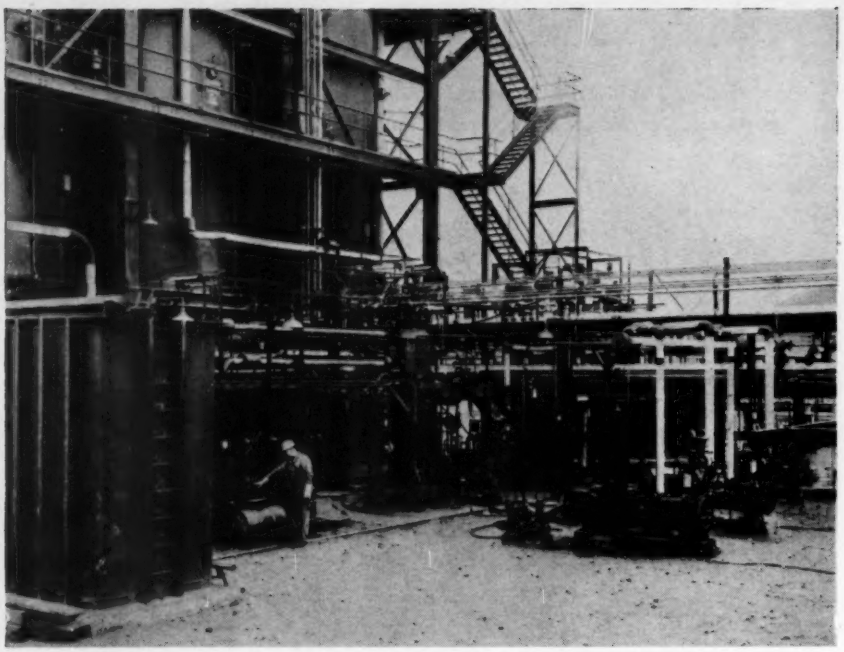
In Germany, in 1913, F. Bergius found that coal can be converted to oil if treated with hydrogen at about 425°C., under pressure of 1,500 p.s.i. Adding oil to the coal to make a paste that could be injected by pumps made it possible to perform this reaction continuously. Started in 1924, this process was further developed in pilot and demonstration coal hydrogenation plants in Germany. Advances in metallurgy during the same period brought forth stronger hydrogen-resistant materials for high-pressure vessels, tubing and pumps.

The first commercial plant was built at Leuna, Germany, in 1926. The process was divided into two stages—liquid- and vapour-phase—and efficient vapour-phase catalysts were discovered. These improvements served to increase the throughput and yields, and made the coal-hydrogenation products comparable in quality with the products from petroleum oils. The first basic raw materials were lignite and lignite tars. Later, crude-oil fractions, bituminous coals and coal tars were used. Twelve hydrogenation plants were built in Germany, none of which supplied appreciable quantities of chemicals. But these plants eventually supplied at least 85% of the German aviation spirit during World War 2, and other products such as motor spirit, diesel fuels, jet fuel, fuel oil, paraffin wax and lubricating oils were produced in quantity.

In England, a bituminous-coal hydrogenation plant was put to work at Billingham by I.C.I. in 1935. During the war, however, the plant switched to creosote oil.

Towards the end of World War 2, the U.S. Government became very concerned about future supplies of liquid fuels, in view of the rapidly increasing demand and the slowing down in the rate of discovery of fresh reserves of petroleum. In 1944, $30 million was voted to the Bureau of Mines for research and development work on synthetic liquid-fuel processes, including coal hydrogenation. In 1948 and 1950, further sums of $30 million and $27 million were allocated for the extension of this work. As part of its development programme, the Bureau has erected a 'demonstration' coal-hydrogenation plant at Louisiana, Missouri, built in part using transplanted German equipment. This plant was completed in May 1949.

In the Bureau's process, coal is liquefied by reacting it with hydrogen under suit-



The high-pressure structure, with some of its auxiliary equipment

able conditions of temperature and pressure in the presence of a catalyst and a recycle oil. The plant was designed to operate at 10,000 p.s.i. pressure. Liquid-phase hydrogenation is accomplished by first pulverising the coal and mixing it with a somewhat larger quantity of heavy oil recycled from the process and a small percentage of catalyst. The paste so formed is pumped with hydrogen through preheaters into a series of high-pressure vessels. Pressure is maintained at some constant value between 7,500 and 10,000 lb., and temperature is held constant somewhere in the range 450 to 500°C. Lighter products of the reaction are separated and further processed in a vapour-phase hydrogenation operation which converts these oils to petrol and other products. The heavy oils from the first separation are recycled for mixing with coal to make paste feed to the first converter stage.

Advantages of new process

Carbide & Carbon Chemicals Co. began their research on the coal hydrogenation process as far back as 1935. The present plant represents a research investment of about $8 million. When, in 1945, work had reached a point where expansion to pilot scale seemed indicated, the company decided against erecting a small-scale pilot plant owing to the comparative costliness of such a plant and the limited return in experience to be gained from it. It was decided instead to build a plant on a semi-commercial scale. While the plant at Institute is therefore nominally a 'pilot' plant, its capacity and potentialities make the use of such a term misleading.

In support of their claim to have converted this uneconomical German process for fuel production to a profitable venture for the production of chemicals, Carbide list the following main achievements in the design of their plant: (1) Coal processing time under high pressure with hydrogen has been reduced from between 45 and 60 min. to 4½ min.; (2) operating pressures have been reduced from between 7,500 and 10,000 p.s.i. to between 3,500 and 6,000 p.s.i.; (3) less hydrogen (a high-cost item in liquefying coal) is required than in the German process; (4) instead of fuels, the products are composed almost entirely of aromatic chemicals; and (5) only one liquid-phase hydrogenation step is included, eliminating the vapour-phase operation entirely.

In addition, the investment required has been reduced appreciably, the refining processes have been simplified so that they can be run continuously and with little operating labour, and engineering design has been refined to such a point that operating difficulties will be a minor factor.

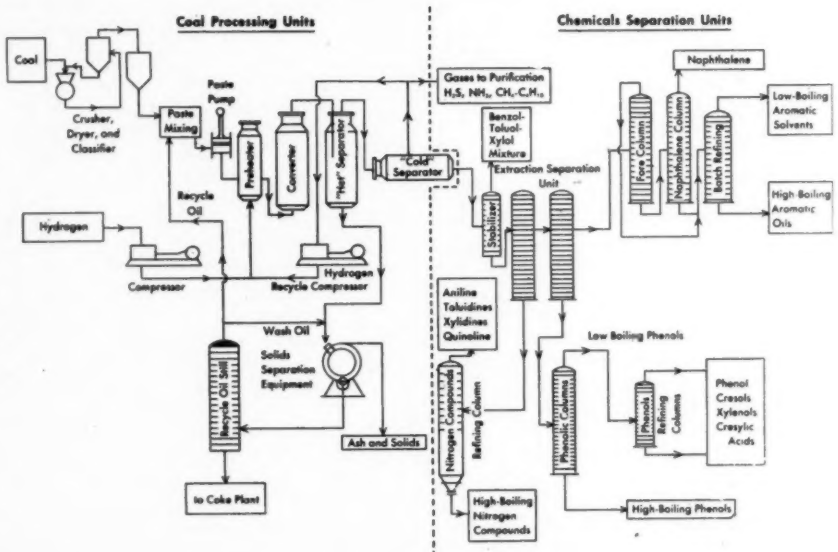
The process yields, on a percentage basis, very much larger quantities of the higher members of the series than the by-product coking of coal. Not only are substantial quantities of benzole, phenol and aniline obtained, but also much larger quantities of their higher homologues than was hitherto possible. For example, 1 ton of coal will produce 5 to 8 times as much naphthalene as the by-product processes, 100 to 200 times as much of the higher phenols such as cresol, and 300 to 500 times as much of the higher amines such as quinoline. Aniline is obtainable in large quantities, whereas in by-product recovery from coke ovens there is no commercial isolation of aniline.

Process and equipment

Spread over a 20-acre site, widely spaced to permit expansion and to minimise hazards, are four major processing structures and three principal control buildings, a substation and several minor service buildings and structures.

Coal from the nearby West Virginia hills is received at the gate-house, located on the highway in the centre of the north side of the site, by truck and retained in temporary storage on the north-east area of the site. From here, coal is bulldozed to a pit on the east side of the storage area, then carried on a belt conveyor and ground in the primary crushing equipment located

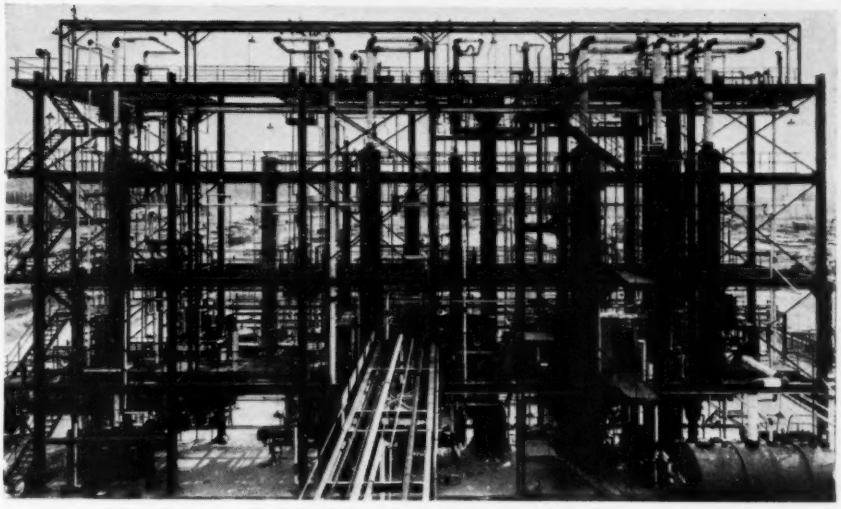
CHEMICALS PRODUCTION by COAL HYDROGENATION
Simplified Flowsheet



at the point of the angle between the two flights of the coal conveyor.

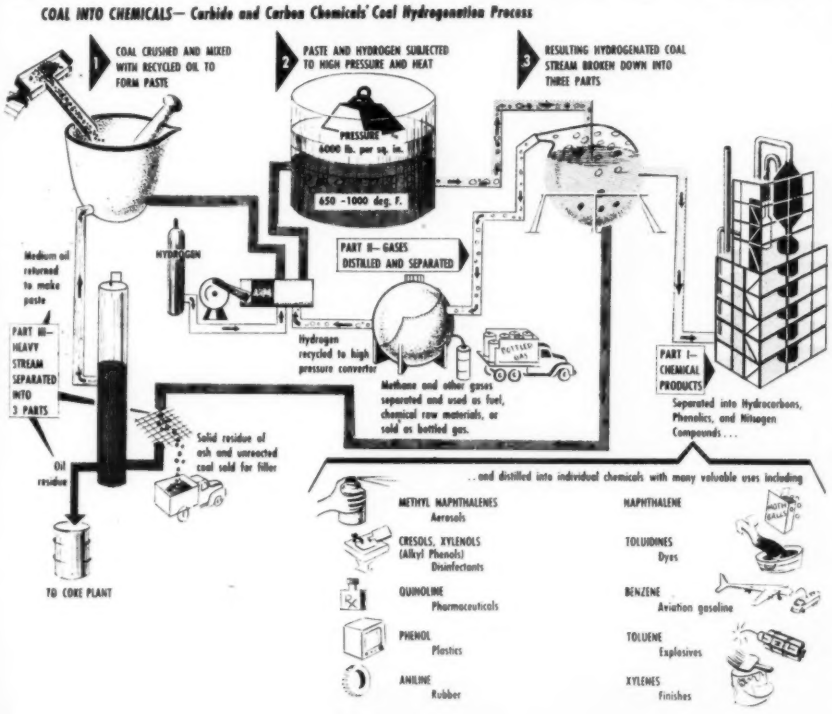
Crushed coal proceeds up the second flight of the conveyor and may again be diverted to temporary storage or continued uninterruptedly to the crushed coal storage hopper. Crushed coal from the storage hopper proceeds north to the coal preparation structure located at the south-west corner of the coal storage area. In this building, coal is further pulverised to about the size of wheat grains and dried in a continuous stream of hot furnace gases. Then the dried, pulverised coal is mixed with a recycled oil, recovered from the process in a later stage, and pumped by the coal paste pumps at high pressure across the roadway west to the high-pressure building.

This high-pressure building is a tall, steel structure supporting three large, vertical pressure vessels each surrounded by heavy-sheet-metal stacks. Heat, as necessary to the process, is supplied to incoming materials in the eastern-most high-pressure vessel, which serves as a preheater. Preheated hydrogen and coal paste enter the high-pressure converter (the centre pressure vessel) continuously. In this vessel, coal paste reacts with hydrogen to form a predominantly liquid product. Since this is an experimental plant, provision has been made to allow process operation at any temperature from 450 to 550°C. (840 to 1,000°F.) and pressures up to 6,000 p.s.i. From the high-pressure converter, the liquid proceeds continuously to the third or western-most vessel, called the 'hot' separator, in the high-pressure building. Here the liquid and vapour streams are separated, following which the pressure of the liquid stream is reduced. The vapour stream is condensed just north-east of the building and continues to a small horizontal pressure vessel, called the 'cold' separator, slightly further west. In the 'cold' separator, gases and unreacted hydrogen are released and returned to the compressor building for recycle to the process.



Chemicals separation unit

At this point in the description we have described how the hydrogenation product has been broken into three streams: (1) a liquid product containing the extremely-high-boiling oils, ash and unreacted coal; (2) a gas stream containing light gases and hydrogen; and (3) a liquid stream containing the chemical products. Let us now follow each of these streams separately.


COAL INTO CHEMICALS— Carbide and Carbon Chemicals' Coal Hydrogenation Process
1 COAL CRUSHED AND MIXED WITH RECYCLED OIL TO FORM PASTE
2 PASTE AND HYDROGEN SUBJECTED TO HIGH PRESSURE AND HEAT
3 RESULTING HYDROGENATED COAL STREAM BROKEN DOWN INTO THREE PARTS
PRESSURE 6000 lb. per sq. in.
650 - 1000 deg. F.
Medium oil returned to make paste
HYDROGEN
PART II— GASES DISTILLED AND SEPARATED
Hydrogen recycled to high pressure converter
BOTTLED GAS
Methane and other gases separated and used as fuel, chemical raw materials, or sold as bottled gas.
PART III— HEAVY STREAM SEPARATED INTO 3 PARTS
PART I— CHEMICAL PRODUCTS
Separated into Hydrocarbons, Phenolics, and Nitrogen Compounds . . .
Solid residue of ash and unreacted coal sold for filler
Oil residue
TO COKE PLANT
. . . and distilled into individual chemicals with many valuable uses including
METHYL NAPHTHALENES Aerosols
CRESOLS, XYLENOLS (Alkyl Phenols) Disinfectants
QUINOLINE Pharmaceuticals
PHENOL Plastics
ANILINE Rubber
NAPHTHALENE
TOLUIDINES Dyes
BENZENE Aviation gasoline
TOLUENE Explosives
XYLENES Finishes

The first or heavy stream from the hot separator containing the very-high-boiling oils, unreacted coal and ash is piped to the heavy-products separation structure located south of the large storage tanks, which are themselves south of the high-pressure building. In this heavy-products separation building, unreacted coal solids and ash are removed from the oil by a solids separation process, which may involve centrifuging, filtration or flash vaporisation. The solids removed from the heavy-process stream in the heavy-products separation building contain the ash of the coal plus some carbonaceous material. This carbon comprises those portions of the coal that do not and cannot be made to react with hydrogen under any reasonable process conditions.

The separated heavy liquid oil is distilled into three fractions in this building. The most volatile fraction is used as a wash oil to purge the remaining heavy oil in the solid residue. The next higher-boiling fraction, which comprises a major portion of the liquid product from the high-pressure hydrogenation, is returned as recycled oil to the paste preparation building to pick up more coal and so return to process. The residue from this distillation is potentially useful for other products used by other Divisions of Union Carbide & Carbon Corporation to supplement supplies of high-carbon-content raw materials.

Picking up next the second stream mentioned above—the gases from the 'cold' separator—these are piped to the compressor building where they are recompressed to process pressure, if sufficiently high in hydrogen, and returned to the high-pressure converter. A portion of these gases containing predominantly methane and fuel gases and some light hydrocarbons, similar to the heavy constituents of natural gas, are returned to Carbide's nearby plants for separation, use as fuel, chemical process raw materials, or sale as *Pyrofa*

('bottled' domestic heating and cooking gas).

The third or chemical products stream originating at the 'cold' separator, mentioned above, contains the principal values in which Carbide & Carbon Chemicals Co. is interested. This chemical stream is piped from the 'cold' separator to the chemicals separation unit located immediately south of the principal process control building which, in turn, is south of the heavy-products separation structure. This stream contains all of the important chemical products to be recovered, including toluol, xylol, naphthalene, methyl naphthalenes and higher hydrocarbons; cresols, xylenols, minor amounts of phenol and higher monohydric phenols, aniline, toluidines, xylidines, quinoline, methylated quinolines, indole and other higher-boiling nitrogen-containing chemicals. In the light-products separation building, this stream is first broken into three principal fractions by chemical reaction and solvent extraction: (*a*) a hydrocarbon portion, (*b*) a phenolic portion and (*c*) an aryl amine and nitrogen heterocyclic portion.

The hydrocarbons are separated by distillation into various saleable products, one of which, naphthalene, is further purified by crystallisation. The phenolic portion is separated by continuous distillation into two fractions, one containing important resin raw materials and the other containing higher-boiling phenolic materials. The crude hydrocarbon fractions and the crude phenolic fractions are further refined by various processes to obtain specification grades of aromatic solvents and resin raw materials. The nitrogen compounds portion is separated by batch distillation to yield aniline, toluidine, xylidines, quinolines and other products. All of these finished products are stored prior to shipment in the product receivers and product storage tanks.

An important feature of the plant, not specifically part of the process itself, is the cleaning of process wastes (carried principally in water) before discharge to the Kanawha River. Two large treatment tanks used to render the sewered water innocuous are located across the roadway near the light-products separation building.

This description has been necessarily brief to avoid confusing detail. Included in the plant are, of course, a large number of tanks for in-process storage, bulk storage of the numerous products, feed tanks, receivers, etc. Not the least important from the economic point of view are the loading platforms and racks for filling tank-cars for shipment of products. These are located south of the railroad spur which crosses the extreme southern end of the process area.

Chemicals from Coke Oven Gases

CURRENT AND FUTURE TRENDS EXAMINED

It is a disconcerting thought that the cost of gas to industry in this country, as measured by heat units, is about four times that of raw coal. Until more economical production is obtained we are not making the best use of our coal resources. One of the most important considerations in arriving at a more economical use of coal is the recovery of by-products from the carbonisation process. Here is an abstract from a paper in which the latest practices in extracting by-products from coke oven gases are described, and some pointers to greater economy are given.

A TABULATION of the by-products obtained from coal during high-temperature carbonisation and a comparison with the yield obtained under low-temperature conditions makes it clear that the highest yield and quality of products could probably only be achieved by some dual process of high- and low-temperature carbonisation. This would permit the isolation of low-temperature gases and tars, followed by the degasification, partially or to the limit, of the coke product to the desirable requirements of domestic or metallurgical fuels.

So far, however, despite several attempts, this has not yet emerged as a practical proposition and recovery of coke oven by-products must, for the time being, continue to be harnessed to high-temperature carbonisation.

With these observations, C. L. Boucher and D. Bagley opened their paper, 'Developments in the Recovery of By-products from Coke Oven Gases,' read recently before the Society of Engineers in London.

Continuing, the authors pointed out that in the modern by-product recovery oven heated by coke oven, blast furnace or producer gas, the coal charge being raised to about 1,000°C. yields as by-products:

Coal tar, fractionated into its several constituents.

Ammonia, either as liquor or processed to sulphate.

Benzole, toluol, xylol and naphthas, and naphthalene.

Stripped gas for heating services.

Phenols may be, and in some cases are, abstracted.

Coal tar. Continuous operation over a protracted period is one of the more noteworthy achievements in modern tar recovery practice—a marked change from the frequent shut-downs necessary with many of the earlier types of plant. This continuity is achieved through the application of certain principles in the metallurgy of the tube still through which, after preheating, the crude tar is pumped. A combined distillation and fractionation plant is employed in which one column is operated at atmospheric pressure and the other under vacuum, both being equipped with side strippers to yield commercial specification products.

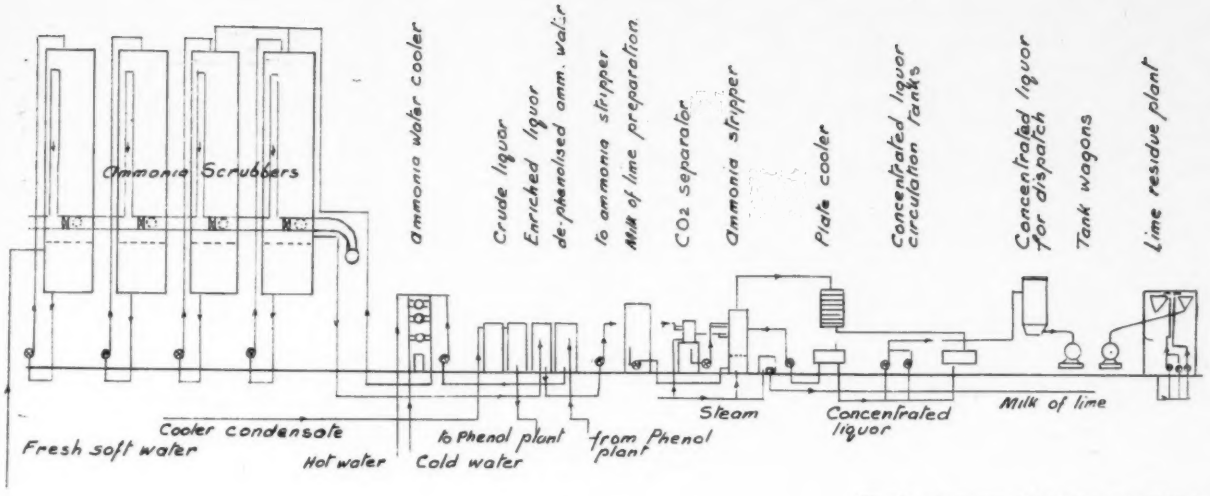
Phthalic acid via the naphthalene is a relatively new feature rendered economically feasible when a contact sulphuric acid plant is available.

Ammonia. The abstraction of ammonia from the gas stream usually follows one of two established methods, the direct and indirect processes. There are several processes for the removal of hydrogen sulphide, apart from the use of iron oxide which may be used to remove the last traces of sulphur from the original gas. Pelleted oxide is the most convenient. Organic sulphur is removed by an alkalised oxide.

Subject to the prior removal of naphthalene, admirable service is rendered by a plate and bell washer, the function of which is to secure the best contact between the scrubbing medium, which for hydrogen sulphide is ammoniacal liquor withdrawn from the plant, and the gases containing the sulphur compounds. These latter are admitted at the lower extremity of the washer and flow upward through the orifices formed in several bells in each superposed plate. These are submerged in the washing fluid admitted by a pipe situated above the uppermost plate. The initial washing fluid may be fresh water, unless it contains an appreciable percentage of lime, in which case cooled waste liquor from the ammonia stripper (which forms part of the ammonia abstraction plant) may be utilised to take up the last trace of ammonia.

In the central portion of the washer, the fluid is enriched by ammoniacal liquor. At this point, 35 to 40% of the hydrogen sulphide is weakly associated with the ammonia; by increasing the ammonia content of the fluid to about 18% on the lower plates, 90 to 95% of the hydrogen sulphide is combined and an appreciable portion of the coincident carbon dioxide is removed. The now partially acidified washing fluid is conducted to a second plate and bell washer, in the lower section of which it is heated to 95 to 98°C., where hydrogen sulphide and carbon dioxide are dissociated and liberated.

At the top of the column fresh water is admitted, and the temperature reduced to



(Flowsheets by courtesy of the Society of Engineers)

Pumps shown thus ⊗

Concentrated ammoniacal liquor plant

a point which prevents the escape of any traces of ammonia. The bell washer is also successfully employed for benzole abstraction.

The mixture of hydrogen sulphide and carbon dioxide may be associated with similar gases and vapours from the ammonia 'saturator,' and then pass to the sulphuric acid plant. The de-acidified ammoniacal liquor is returned to the ammonia stripper and thereafter used in the normal manner for the production of ammonium sulphate or concentrated ammoniacal liquor.

Sulphuric acid. The heavy and expanding demand for sulphuric acid and the coincident shortage of sulphur have naturally turned the thoughts of chemical engineers to the production of this acid from sulphur-containing gases.

Average coke oven gas contains about 500 gr./100 cu. ft. of hydrogen sulphide, which can be burnt to sulphur dioxide and from which sulphuric acid is ultimately produced.

Several methods exist by which hydrogen sulphide can be removed from coke oven gas and of these the *intensive tower system* earns distinction. The great advantage of the process lies in the simple, durable and economic design of the towers.

The incoming mixture of hydrogen sulphide and carbon dioxide from the ammonia plant is burnt in a furnace, forming sulphur dioxide and water. The heated gases are passed through a waste-heat boiler, during which they are cooled to about 350°C., at which temperature they pass to the towers where the further oxidation of the sulphur dioxide is effected. Finally, to obtain an end product entirely free from nitric acid, the sulphuric acid is pumped back to the denitration and dehydration tower.

In sulphuric acid plants generally, efficiency and economy are influenced by the ratio of the product to the volume of packed space in the various towers. Obviously, then, the design of the towers should be such as to achieve the greatest ratio of output to packed space, thus reducing the overall cost and enabling comparative small outputs to be obtained economically. In all production processes, it is desirable to be able to vary the output without undue loss of efficiency. In the system described the rating is −50 to +25% or more as may be required.

Benzole. Crude benzole is recovered from the coke oven gas after it has been stripped of tar, ammonia and sulphur, and is subsequently rectified to bring it within market specifications. No drastic changes have been introduced in benzole recovery methods in recent years. In general, the scheme of benzole recovery may be stated as follows: (1) the extraction of benzole homologues from crude coke oven gas by bringing the latter into intimate contact with a suitable absorbent maintained at a low temperature; (2) separation of this mixture from the absorbent; (3) fractionation of the benzole mixture into groups of definite boiling point limits; and (4) rectification of the fractions to bring them within the required industrial specifications.

Methane. Methane is usually present in coke oven gas to the extent of about 30%. Its gross calorific value is relatively high, being about 8,940 Kcal. cu.m. or about 1,050 B.Th.U. cu.ft. If by abstracting and liquefying 90% of the methane resulting from the carbonisation of a ton of coal the equivalent of 21 gal. of petroleum spirit could be actually recovered, such a procedure would clearly be attractive. Unfortunately, this idea cannot be realised simply, since liquid methane cannot exist under any pressure at a temperature above −82°C.

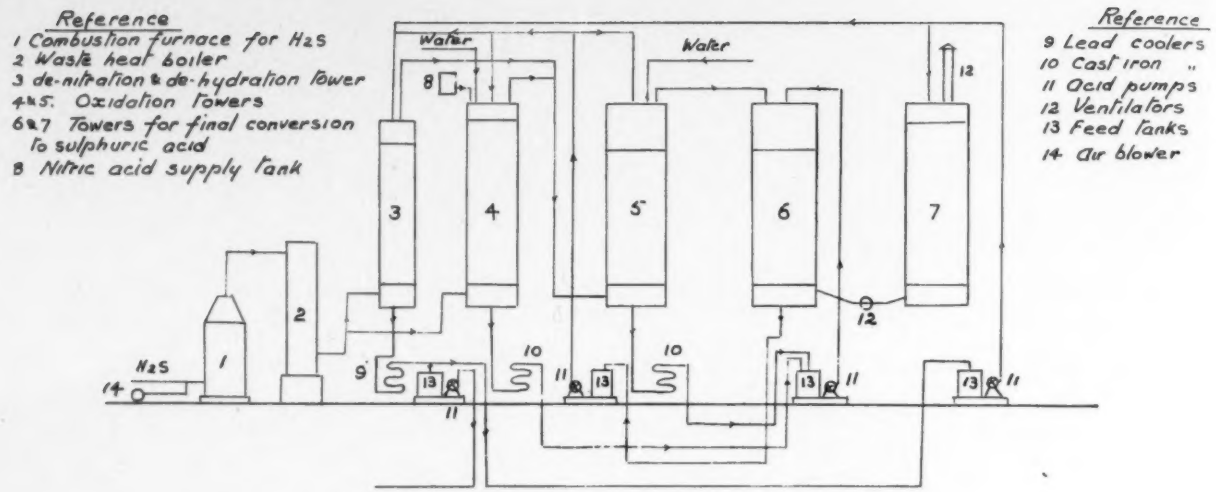
By a catalytic process, methane gas may be converted to the higher members of its series liquid at atmospheric temperatures, an adaptation of the Fischer system. As regards the quantity available, it has been estimated that the equivalent of some 400 million gal. of petrol yearly are potentially recoverable from coke oven gas alone (based on the carbonisation of 20 million tons of coal annually), though this would be at the expense of certain other uses for the gas.

A pilot gas-liquefying plant was erected some time ago in the north of England. Here, on the large scale, the raw gas already stripped of tar, ammonia and sulphides, is cooled to about −40°C., so liquefying benzole products and avoiding the use of absorbent oil, only fractionation being subsequently required. The gas is then reheated in counter current to about 10°C., compressed to about 8 atm. and passed through pre-cooled metallic element regenerators, reducing the temperature to about −132°C., where it is expanded through a turbine which lowers the temperature still further to a point where the methane liquefies and is withdrawn. The cold gases ascend another regenerator, so cooling the metal elements therein. They are returned to the system at a temperature of about 5°C.

Possible future developments

The authors closed their paper with the following comments.

It is a disconcerting thought that the cost of gas to industry in this country, as measured by heat units, is about four times that of raw coal. The difference is too great, and that it should exist is in itself an economic disadvantage that seems to call for drastic amendment in the entire system of production. Until a much better thermal efficiency is obtained, with a consequent reduction in labour costs, combined with a rational heat treatment of the raw coal permitting the production of solid fuels suitable for domestic and industrial purposes, we are not making the best use of our coal resources.



Intensive tower plant for manufacture of sulphuric acid

An interesting sidelight on the present state of affairs is afforded by the grid prices received in 1952 for the surplus gas from metallurgical coke plants, which was of the order of 3d./therm for unpurified gas. It is doubtful if gas works costs for comparable gas are less than 9d./therm. Under these circumstances, metallurgical coke produced in wasteful beehive-type ovens, without recovery of by-products, might form an investment superior to the modern coke oven.

What is the answer to this? While the complete path ahead is not clear, there are a few direction signs which, if followed, might lead to the desired goal.

When coal is distilled at about 400°C., the gas evolved contains about 20% of hydrogen, 60% of saturated hydrocarbons and 6% of unsaturated hydrocarbons. If the distillation temperature is raised to 500°C., the hydrogen fraction is increased, and the saturated and unsaturated hydrocarbons are diminished. The calculated calorific values of the gases evolved at 400 and 500°C. being 6,800 and 6,600 Kcal./cu.m. respectively and the volumes 140 and 160 cu.m./ton of coal respectively. At the higher temperature there will be about 20 gal. of tar oils per ton.

If the temperature of the resulting coke is now raised to 1,000°C., a further 150 cu. m. of gas is evolved with a calorific value of only 3,500 Kcal./cu.m. (or about 400 B.Th.U./cu.ft.), the hydrogen content rising to over 70% and that of the saturated and unsaturated hydrocarbons declining to 20%.

The coke resulting from distillation at the 500°C. temperature obviously contains the volatile matter driven off at the higher, and is thus more easily ignited, yielding a fuel for open grates with much higher radiant energy than raw coal, though unsuitable for metallurgical purposes. The two types of gases can be combined and form a high-grade gas.

Continuous distillation in vertical retorts typifies this mixed gas, as about 60% of the coal is distilled under L.T. conditions, and some of the sensible heat of the coke is recovered. The tars from this system represent an improvement on the usual run of coke oven tars.

So far the impossibility of effectively separating the L.T. and H.T. gases under any system of commercial value has prevented the undoubted benefit accruing from high yields of tar oils suitable for hydrogenation into motor fuels, phenolised substances for the plastics industry, etc. A two-stage system with this objective is, however, being developed, and this may be regarded as one signpost.

From a gas production aspect, the complete gasification of coal under pressure is another possibility, though clearly of no use where solid products are one objective.

Other directions in which potential economies exist lie in the recovery of the sensible heat of the coke for further use in the carbonising process, or elsewhere. In coke oven practice, it is usual to quench the coke with water (sometimes with the effluent from the ammonia stills), in this way sacrificing 350 Kcal./kg. of coke. In quenching about 30 million tons of coke ex coke ovens and gas works, this represents a loss of about 1½ million tons of coal yearly.

These are disturbing thoughts, but in the opinion of the authors, it is along these lines that improvements and economies will come, and enable us to conserve our heritage of coal.

Solvents Produced in Canada

(*Concluded from page 164*)

Acid flow measurement

The type of instrument used for measuring the flow of hot dilute acid to the Chemico concentrator may be of interest to those faced with the problem of metering such streams. A *Magnabond*-type rotameter is used. The body of the meter is of lead-lined steel protected by impervious graphite. A carbon metering tube houses a *Teflon* float, with the metering edge of tantalum. The float extension rod is made of *Pyrex*-glass-covered *Hastelloy*.

The Montreal chemical plant embodies numerous operational and equipment refinements developed by Shell during almost 20 years of experience in the operation of the basic steps of this process in the U.S. and in the United Kingdom. The highest possible degree of chemical engineering design and the strictest adherence to high safety standards make this plant as safe and as modern as any in the petrochemical field.

The C.F. Braun Co., of Alhambra, California, was engaged for equipment layout, design, fabrication and field erection. Process design was by Shell.

Plastics moulding. R. H. Windsor Ltd. have produced a number of new illustrated leaflets describing their latest plastics moulding machines and extruders. Included among these are the latest 1-oz. automatic injection moulding machine, the *SH* 2-oz. and 8- to 10-oz. injection moulding machines, and the *RC* 100-lb. extruder, all of which were on show at the B.I.F. in Birmingham and described in our May issue. Other machines described in these leaflets are the *SH* 3-oz. injection moulding machine, a hydraulically-operated machine driven by a self-contained electric motor; the *SH* 4-oz. machine, which is fully automatic and has been designed to provide a medium-capacity machine capable of producing almost any article in a wide range of material; the *SH* 16/20 and 28/32, the two largest moulding presses in the Windsor range; the *RC* 65 twin-screw extruder with variable-speed drive; and the Windsor rigid PVC tube take-off equipment. This last is a compact unit consisting of extruder, die, water-cooling bath, pulling device and cut-off saw which, the manufacturers claim, will produce pipe and tube to the closest tolerances known in the industry today.

CRYSTALLISATION

Ammonium sulphate; ammonium nitrate; salt production; crystal growth; crystallisation processes and techniques

By A. W. Bamforth
(Power-Gas Corporation Ltd.)

Ammonium sulphate from petroleum residues

NOTWITHSTANDING the great advances made in methods of treating petroleum for the improvement of colour and odour and the removal of sulphur, and the fact that acid treatment is not well suited for the refining of cracked spirit, the original refining method using sulphuric acid is still widely employed and is a most effective one. Sulphuric acid treatment, however, created the problem of disposing of the acid sludge produced. Now a method has been developed[22] for disposing of the waste without causing atmospheric pollution, while simultaneously converting the formerly waste materials into usable products.

Acid sludge stock containing 41% sulphuric acid and 45% oil is neutralised with a foul steam containing 2% ammonia and 2% hydrogen sulphide. The mixture of ammonium sulphate and oil is continuously separated; some light oil is added and separation is readily achieved on account of the high gravity of the sulphate liquor. The oil portion is transferred to stock tanks and can be used as fuel oil.

Ammonium sulphate liquor from the separator at approximately 110°C. is introduced into a Krystal crystalliser where concentration and subsequent crystallisation takes place by evaporative cooling. It is of interest to note that if the sulphuric acid content of the oil sludge is more than 30% evaporation can take place in the crystalliser without the addition of heat from other sources. If the sulphuric acid content is low, then the crystalliser system must be fitted with a heater of adequate capacity to supply the additional heat required for evaporation. The flow sheet of this process is shown in Fig. 1.

A method of purifying ammonium sulphate produced from ammonia and spent petroleum refinery acid by washing with volatile solvent has been patented.[67] The solvent, which is water soluble, dissolves the organic impurities, but is a poor solvent for the ammonium sulphate. The process can be applied to moist salt and so serves to dry the salt by the solution of the water as well as the impurities in the water.

The effluent liquors from DDT manufacture contain considerable amounts of sulphuric acid and chlorobenzenesulphonic acid. This waste liquor is mixed with an aqueous solution of ammonium sulphate and cooled, giving rise to separation of chlorobenzenesulphonic acid as crystals. The clear filtrate is neutralised with gaseous ammonia and then cooled to crystallise ammonium sulphate. The filtrate from this second separation is recycled.[18] Ammonium sulphate is also recovered from waste sulphuric acid and commercial ammonia.[51]

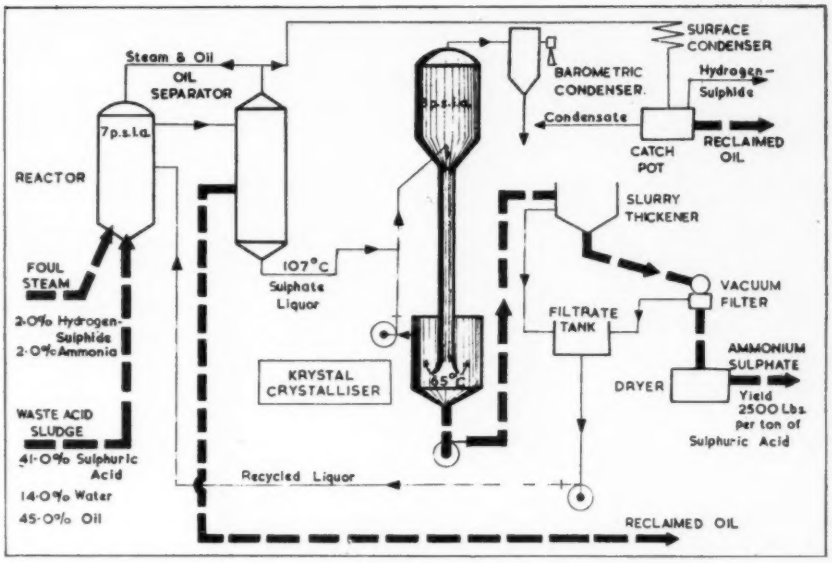


Fig. 1. Flowsheet of system for the recovery of ammonium sulphate and oil from petroleum refinery acid sludge

Refined ammonium sulphate is crystallised from solution from which iron, lead and heavy metals are precipitated as hydrates or sulphides.[71] The treatment of saturated ammonium sulphate solutions with a soluble fluoride in order to precipitate iron and aluminium impurities and to accelerate the formation of ammonium sulphate crystals has been patented.[64]

Ammonium sulphate has been produced on a semi-industrial scale by the oxidation of ammonium sulphite solution with air or oxygen without the use of catalyst.[29] A further technique for making ammonium sulphate by the sulphurous acid method is described by Miyama[40] and a similar process is patented by Collier.[15] Ammonium sulphate, together with potassium sulphate, has been produced from iron potassium sulphate ore by heating the ore to 700°C. and treating with ammonia gas.[58] A process has been patented[1] for obtaining ammonium sulphate from gypsum by the treatment of an aqueous solution of gypsum with ammonia gas and carbon dioxide.

Ammonium nitrate

Pilot-plant work by Miller and Saeman[39] has led to the construction of a 525-ton/day Krystal ammonium nitrate crystalliser plant by the Tennessee Valley Authority at Wilson Dam, Alabama. This plant provides a safer and more economical method of producing ammonium nitrate than the graining process. It was put into full-scale operation in 1950. An article describing the plant, which is the first of its kind to use continuous vacuum crystallisation for the large-scale production of this salt, has been published.[53] Certain features of the process have been patented.[52]

The crystallisation plant, consisting of five vacuum crystallisers in parallel, is

operated above 90°F. to produce form III crystals because of the greater steam economy in vacuum operation and because of the greater difficulty in drying form IV crystals. The product consists of single rounded crystals predominantly −16 +35 mesh in size. By a slight modification in operating technique, crystals of up to 6 mesh can be produced at the rate of 200 tons/day.

The crystals are centrifuged, dried and treated with about 4% of −200 mesh clay. The bagged crystals are said to store and spread well.

Treatment of ammonium nitrate and ammonium nitrate explosives with acid magenta to prevent caking in storage is fully described.[66] The addition of this dye alters the crystal structure of ammonia nitrate IV.

Ammonium nitrate, whether treated with clays, etc., can in storage under certain conditions become caked in one solid mass, due to the cementing together of the crystals by similar crystals which recrystallise from the saturated solution which moisture has formed. The effectiveness of acid magenta lies in the fact that if caking occurs the bond between the normal crystals is modified into the form of soft, extremely friable flakes, and is easily broken down in normal handling. The dye colours the product, but this has no commercial disadvantage. It has not been possible to find dyes to give the same effect as acid magenta on nitrate III or nitrate IV if this is at a temperature above 32°C.

Salt production

What is claimed to be the largest and most highly mechanised solar evaporation salt farm in the world has been described.[9, 54] Prasad[47] describes another experimental salt farm on which two types of salt have been manufactured by using a multiple irrigation system.

Moscu[41] studies the production of solar salt in some detail. Equations are given for the calculation of evaporation, depth of solution and area required. The requirements for tank bottoms are also given, covering such points as impermeability, mechanical strength to permit recovery of the product, chemical composition to avoid contamination and the type of surface required to promote crystallisation. Experiments carried out on similar lines, especially with a view to minimising gypsum deposition, are reported by Lepez.[36] The recovery of sodium chloride from tannery salt by the addition of sodium carbonate is also described.[17]

Three new salt production methods are reported in the literature.

In the manufacture of salt, vacuum pan and grainer pan processes are integrated to give large economies in heat consumption by the full application of vapour recompression.[19] Preheated brine for grainer pan evaporation is concentrated by reducing the pressure to flash off some of the vapour, which is compressed together with vapour from the vacuum pans by partial use of actuating steam. All the steam thus provided is used in evaporating additional brine in the vacuum pans and in preheating brine for the grainer pans.

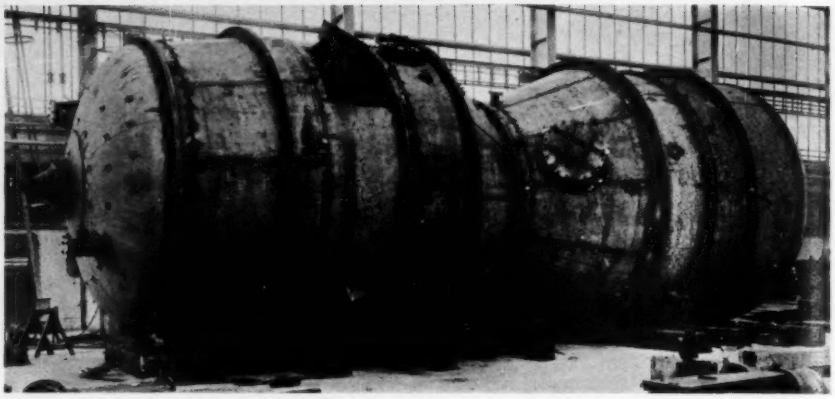


Fig. 2. A 15 ft. 6 in. diam. rubber-lined mild-steel 'Krystal' sodium chloride evaporator undergoing hydraulic test at the Power Gas Corporation's works at Stockton-on-Tees

Krystal evaporators for the more economical production of a coarse-grained salt which is steadily superseding pan salt is described.[3] This salt is produced from well brine heavily contaminated with sodium sulphate. A Krystal evaporator, which is 15 ft. 6 in. outside diameter and 40 ft. long, is shown in Fig. 2. The calandria tubes on this apparatus had only an eggshell deposit of scale after two year's operation.

As a development on the foregoing, a triple-effect Krystal plant has been designed in which the gypsum impurity is separated in the solid phase by controlled crystallisation simultaneously with sodium chloride, consequently the costly chemical treatment plant which is normally installed to precipitate calcium is not required. Parts of this triple-effect plant, which is constructed mainly in solid nickel, are shown in Fig. 4.

A new development by International Salt Co. for refining rock salt or recrystallising vacuum salt to obtain salt of higher purity is described by Richards.[49] Vapour recompression is used and under suitable conditions there is sufficient heat present in the solution to give evaporation without any supply of external heat; consequently there are no heating surfaces on which scale can deposit. The process is, of course, not applicable to the evaporation of well brines.

Methods of scale prevention in crystallisers and evaporators are mainly in the form of palliatives, *e.g.* the coating of the inside of the crystalliser with chromium, paraffin or bitumen[68] or dissolving or melting the crusts by means of hot air or steam[62] or the addition of scale preventives. Ulmer and Caro,[61] experimenting with beet sugar juices, found that, although treatment with various types of commercial scale preventives improves evaporation rates to different degrees and reduces scaling, no available physical or chemical treatment will completely prevent scaling in sugar evaporators in all locations at all times.

Crystal growth

The effects of nearly 500 different dyes on the crystal growth of potassium salts, ammonium chlorate and borax have been tabulated.[6] The relative effectiveness of the dyes in causing habit modification was measured in terms of the weight in inorganic substance modified by a standard weight of dye. Pleochroism and growth inhibitors are briefly discussed.

Crystallisation of xylose syrup is greatly accelerated by the addition of 0.5% calcium acetate.[42] Calcium formate produces a similar effect. Calcium sulphate hinders the initiation of crystallisation, but once started the process is more rapid. Magnesium salts have no effect.

The effect of increasing the structural viscosity by the addition of agar-agar on the crystallisation of potassium chloride-potassium chromate, and potassium and sodium nitrates in ternary systems, is to favour dendritic growth of one of the eutectic phases, but does not change the character of the crystallisation process.[1] Sodium carbonate crystallised from saturated solutions containing small amounts of salt of alginic acid is in the form of plates.[69]

Factors governing the rate of crystallisation of supercooled molten TNT have been examined[45] and it is found that a large number of compounds each at a concentration of about 0.02 mole/mole TNT decrease the crystallisation rate. Immiscible compounds have no effect.

McLachlan, *et al.*,[37] have discussed some factors in the growth of crystals. Their report covers extensions to the Donnay-Harker law; dendritic growth; growth of snow crystals, etc.

Krueger and Miller[34] have made experimental investigations of the concentration field during the crystallisation of sodium thiosulphate from supersaturated solutions. A graphical presentation of the positions of the growing faces versus time indicates

in all cases a uniform rate of growth with occasional abrupt changes. These discontinuities are in general correlated with changes in the perfection of the growing face.

Boric acid additions in the order of 0.2 to 0.6 g./l. decrease the rate of crystal growth of single crystals of ethylene diamine tartrate and affect the type of crystal formed.[5] Kolarov[33] has studied the crystallisation velocities of supersaturated solutions of strontium and calcium sulphate. The crystallisation velocity depends upon the *p*H value of the solution. Additions of citrate, acetate and chloride ions diminish the crystallisation velocity, but nitrate and bromide ions increase the rate slightly.

The rate of crystal growth of sodium and potassium acetates, and potassium and ammonium nitrates, in saturated aqueous solutions has been investigated.[8] The rate of growth passes through a maximum only in the case of acetates which produce highly supersaturated solutions.

Sveshnikova[59] has investigated the solubility of the reciprocal system calcium sulphate-potassium chloride-water at 55°C. The solubility of sodium nitrate and nitrite in water in the presence of sodium has been investigated by Kuzminykh.[35]

A review of the field of nitrogenous nucleation of supersaturated inorganic salt solutions has been published[60] as also has an article on the nucleation of sucrose solutions.[63]

The effects of crystallisation of ammonium nitrate and sodium chloride on drying of single drops in quiescent air is described by Ranz and Marshall.[48] It is shown that soluble substances concentrate to saturation on the surface of the drops, irrespective of the average solids concentration in the drops.

The spontaneous crystallisation of potassium chloride, potassium nitrate, potassium chlorate, ammonium chloride, potassium sulphate and sodium thiosulphate from supersaturated solutions has been studied by Srikantan.[57] The difference between the saturation temperature and the temperature at which spontaneous crystallisation occurred is reproducible and characterises the compound, and is dependent upon the temperature coefficient of the solubility.

A study has been made of the effect of fluid velocity on the rates of growth of single crystals of copper sulphate pentahydrate and magnesium sulphate heptahydrate.[27] The rates of growth coefficients of these crystals were found to depend upon the mass transfer coefficient, which varied with fluid velocity, and the rate coefficient of the surface reaction, which varied with temperature. The results illustrate a means whereby available data on mass and heat transfer may be applied to crystallisation problems.

Data on Young's modulus, apparent elastic limit and modulus of rupture have

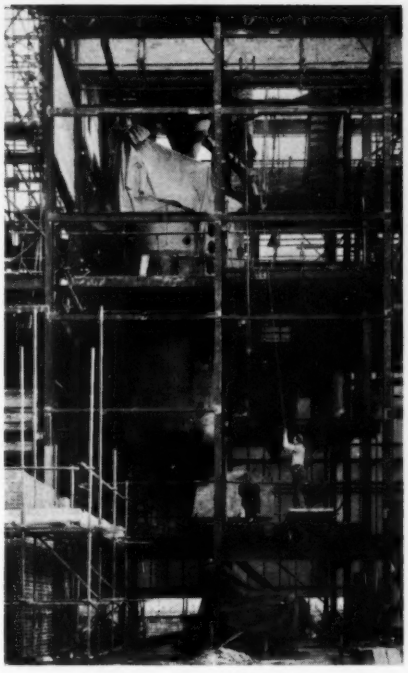


Fig. 3. One of a pair of 'Krystal' rubber-lined crystallisers in course of erection

been obtained from sodium chloride, silver chloride, lithium fluoride, potassium chloride and potassium bromide.[16]

Crystallisation processes and techniques

Separation of rock salt and other attendant salts of potassium chloride is made possible by flotation.[2] The loss of potassium chloride by crystallisation is avoided by keeping the temperature of the slurry constant to prevent the crystallisation of fine potassium chloride crystals on the surface of the removed solids.

Apparatus and process for crystallising and separating, in the form of a slurry, slightly soluble substances from relatively large amounts of liquid has been patented.[43] The process is said to be especially applicable to the production of potassium from sea water in the form of a salt of a highly nitrated aromatic amine such as dipicryl amine.

Urea selectively forms crystalline adducts with straight chain types of compounds, while thiourea selectively forms similar complexes with branched chain and certain ring-type compounds. Once the crystallisation complex is formed, it is removed from the reaction mixture and the crystals are dissolved in water to destroy the complex, the reactant then being recovered as an immiscible layer. The crystals can also be broken down by heat. Kobe and Domask discuss theoretical basis,[30] thermodynamic considerations[31] and practical applications[32] of this process. Fetterley[20] has patented the process of separating organic compounds by crystallisation with urea.

A new continuous evaporative crystallisation process is described[11] which involves blowing an inert gas such as air through a saturated solution. The air evaporates water from the solution and also cools it, so that crystallisation takes place. The crystals are held in suspension by the air flow. The process has been patented.[50] Research work on itaconic acid produced by dehydration or pyrolysis of citric acid or by the fermentation of glucose, indicates that the crystallisation of the acid from concentrated solutions is the best recovery method.[46] A process for the separation of mixtures of compounds by utilisation of a difference in melting points has been patented.[24]

A process has been developed for the separation of stearic and oleic acids by crystallisation from methanol.[12] In this process, mixed fatty acids are dissolved in methanol. The solution is cooled and crystallised at 10°F. in a crystalliser consisting of a bank of jacketed concentric tube heat exchangers fitted with rotating scrapers. The crystallised stearic acid is filtered off, washed and melted before removal of the solvent by distillation.

A fractional crystalliser for obtaining high-purity benzene from a benzene-heptane feed has been patented[56]; it has replaceable rings for scraping the solid material from the inner wall of the crystalliser. A similar apparatus for the same application is also patented,[65] the main feature of which is a porous piston conveyor which does not become bridged-over by the formed crystals. Also described[21, 41] is a continuous fractional crystalliser which claims increased efficiencies, improved separation of mixtures and recovery of high-purity compounds.

So successful has been the Krystal crystalliser installed in 1947 by Eastman Kodak for the production of silver nitrate that a new unit has been built.[10] This process is said to give big improvements over the old batch system, which, at a production rate of 20 lb. per crystallising dish, required hundreds of dishes each needing to be manually handled several times a day. One Krystal crystalliser 4 ft. in diameter and 10 ft. high produces 5 tons/day continuously with only casual attention.

A small all-glass apparatus has been developed[38] which produces crystals of 5 mm. dimensions in 1 hr. It requires only 100 c.c. of solution and can be dismantled for cleaning, having solid material added or solvent extracted without interruption of the process.

A general description of industrial processes for making large single crystals has appeared.[13]

A description of a furnace used and the experimental procedure adopted for the preparation of single sodium nitrate crystals of definite orientation and weights exceeding 1 kg. has been briefly outlined.[70] A method for growing moderately-sized clear crystals of lithium sulphate has been patented.[7]

An apparatus primarily adapted to produce crystalline alumina hydrate from sodium aluminate, but also adaptable to the production of magnesium hydrate from sea water, is covered by a new patent.[25] Crystallisation rates are controlled to give a well-developed granular product. Further research work on the crystallisation of aluminium chloride in the hydrochloric acid process for the production of alumina from clay has been carried out by Clark, Hubbard and Hoffman.[14] Crystallisation was done in a glass-lined evaporator at 124°C. Optimum liquid/solid slurry concentrations are given. This work is a continuation of the development work on aluminium production from clay by the same authors.[28]

A process particularly applicable to concentrated milk products with a seeding surface of lactose crystals has been patented,[55] as also has the production of sodium sulphate from Glauber salts.[26]

A comprehensive review of crystallisation is given by Grove and Gray,[23] who include a larger number of references.

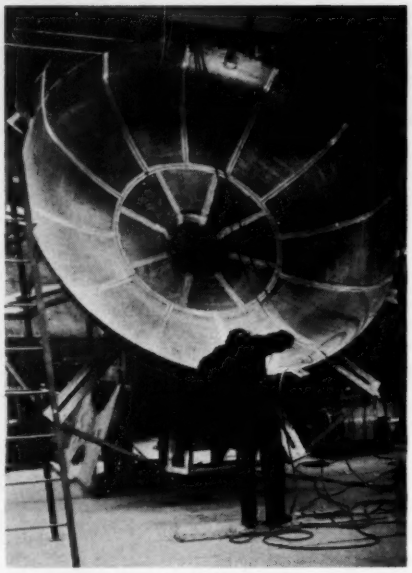


Fig. 4. The segmental dished-bottom part of a solid nickel 'Krystal' sodium chloride evaporator being welded together at Parkfield works, Stockton-on-Tees

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Quality control. The work of the Quality Control Organisation of Davey, Paxman & Co. Ltd. is described in an article reprinted from the October 1952 issue of the *Indian Textile Journal*, Bombay, whose special representative visited the Colchester works of the company. The objects of the organisation are not purely to detect and reject faulty work, but to institute, wherever possible, preventive action by the introduction of tests and checks on raw materials and during all stages of manufacture in order that corrective action can be taken at the earliest possible stages of manufacture. The type of inspection employed by this factory is in the form of combined floor and central inspection, with the maximum amount of floor inspection, as close to the point of production as possible. The system involves statistical quality control, the inspection of incoming raw materials and components supplied by specialised manufacturers, a dimensional inspection department provided with a specially equipped standards room, close control over all inspection and workshop gauges and the systematic sampling and analysis of all foundry materials. In addition, machine-shop production and the assembly of components and engines are carefully supervised. All defects reported, whether from service or during any stage of manufacture, are investigated in detail by a special section. The spares and service departments are organised with similar thoroughness.

Production and Uses of GERMANIUM

By J. A. Gay

(O. W. Roskill, Industrial Consultants)

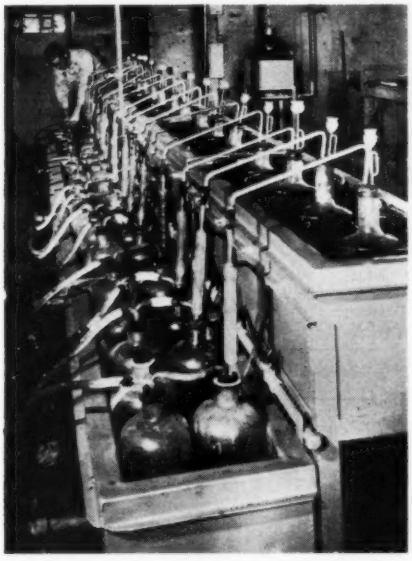
The demand for germanium has risen sharply in the last few years, primarily because of its use in the manufacture of new types of diode and triode valves. Germanium valves require no heater filaments; they are smaller and more robust than those with glass envelopes and have an appreciably longer life than thermionic valves. Germanium diodes are already being used in some television sets, while the germanium triode is likely to be especially useful where space and heater current are both at a premium. Very pure germanium, usually containing less than 1 part impurity in 10^7, is required for electronic uses. The production of germanium of this purity has stimulated work on other applications, including fluorescent lighting, infra-red spectroscopy and metallurgy. Before the recent war, most germanium was extracted from germanite found in south-west Africa, which usually contained 5 to 10% germanium. Since the war, it has been extracted from zinc residues in the United States, and in Britain from gas works' flue dust. So successful has the latter process proved that American companies have started to survey gas works in the United States to see if commercial quantities of germanium are also available there. Although it cannot be said that a boom in the metal is imminent, germanium has obvious strategic uses and it is likely to grow considerably in importance over the next few years.

Production of germanium

WINKLER originally discovered germanium in the very rare mineral, argyrodite, which is an ore containing some silver, germanium and zinc. Shortly after the end of the 1914-18 war, germanite was discovered in the copper mines at Tsumeb, in south-west Africa. This mineral is mostly composed of a copper sulpharsenide which contains some 5 to 10% of germanium. Between the wars most germanium, in Europe at any rate, was extracted from germanite shipped from the Tsumeb mine and, although shipments from this mine ceased during the war, a little germanium is still being extracted from stocks of germanite held in European countries, mainly Germany.

In the United States small quantities of germanium occur in many zinc ores and they have become the main commercial source of germanium there.

Just before the last war Sir Charles Morgan and Dr. G. R. Davies[1] found that flue dust from certain British gas works contained relatively high concentrations of germanium. Subsequent work showed that these metals were present in coal from certain coalfields, particularly those in Northumberland and Durham. This research programme was suggested to Morgan and Davies by the work of Prof. V. M. Goldschmidt who, in 1933, stated that he had found that some coals from the Yard Seam of the Northumberland and Durham coalfield contained some 30 to 50 p.p.m. of germanium. The Fuel Research Station of the Department of Scientific and Industrial Research has recently carried out analyses of coal from all the major coalfields of the U.K. and has found that on the average the quantity of germanium present in coal was much lower than initially suggested by Goldschmidt. Goldschmidt's work was repeated and ten samples of coal from the same seam have been analysed and found to contain from 2 to 22 p.p.m. of germanium, with an average content of about 7 p.p.m. In the last few years about 200 samples of coal from the main coalfields in the U.K. have been examined and it was found that coals from the Scottish coalfields contained 6 to 9 p.p.m. of germanium, Northumberland and Durham 8 p.p.m., Lancashire and north Wales 6 to 9 p.p.m., Notts and Derby 6 to 9 p.p.m., Lancashire and Cheshire 6 to 8 p.p.m. and south Wales 2 p.p.m. It is therefore concluded that appreciable quantities of germanium are present in all the major coalfields in the U.K., except that of south Wales. It is thought that germanium-bearing coals are widely distributed within the coalfields and not localised in comparatively few spots of particular seams. Although insufficient samples have been examined to ensure reliability of results, it is thought that the average germanium content of coal from the Notts and Derby coalfield is possibly higher than that of coals from the Northumberland and Durham coalfield. Work by the Fuel Research Station so far suggests that vitrains may contain more germanium on the average than the surrounding coal, but again too few samples have so far been investigated to verify this.



Primary distillation of crude germanium tetrachloride from hydrochloric acid solution is carried out in the apparatus shown.

The germanium is thought to be associated with the carbonaceous material in the coal rather than with another mineral. This partly explains why germanium-bearing flue dust is more likely to be found in the flues of producer plants than in flues leading from the ovens where the coal is initially carbonised. The average percentage of germanium in dry coal ash produced from dirty coals is 0.006%, while the percentage in ash from clean coals is about 0.013%. As the enrichment factor of germanium content in flue dust to that in the ash is of the order of 12 to 1, the quantity of germanium in flue dust produced from dirty and clean coals should be 0.072% average and 0.156% average, respectively. It is likely that the enrichment factor is generally greater than 12 to 1, as some deposits of flue dust have been found which contain up to 2% germanium.

K. V. Aubery and L. Horton[2] describe the distribution of minor elements in three vitrains taken from the Barnsley seam.

The spectroscopic examination showed that some 18 minor elements were present in the vitrain and that they were predominantly associated with the organic matter in it. It was also found, however, that the relative abundance of these minor elements differed from that of elements occurring in plants living today. These differences may be due to a modification in the plant's mineral demand which has come about since the time when the vegetation from which the present coal has been formed was growing. Germanium is now thought to be one of the minor elements which, although now not present in vegetation, was once absorbed by plants.

Similar investigations into the mineral content of coals have been carried out on the Continent, and it has been found that the germanium content of coals from the Ruhr, Belgium and northern France are considerably lower than that of the U.K. coals. Some coals from the Commonwealth have also been investigated, but very little germanium has been found in them.

It has recently been announced that the Pennsylvania Coal & Coke Co. intend to investigate coal seams in the Appalachian area in the hope of finding recoverable quantities of germanium.

Systematic investigation of coals in Russian coalfields has been carried out and as early as 1943 an account was published[3] about the determination and extraction on a laboratory scale of germanium from coal and industrial waste. Ash from the vitrains of Caucasian coals has been found to contain from 0.1 to 1%, so that if this is true the germanium content of flue dust obtained from this coal may be considerably higher than in this country.

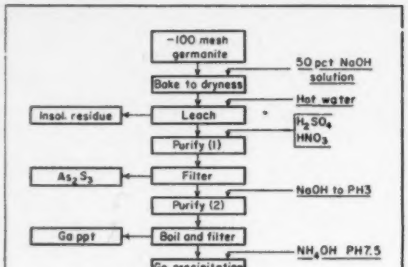
It has recently been reported that germanium has been found in ammonia liquor from the Tokyo Gas Co. works and that a pilot plant is being set up to extract 500 kg. of germanium p.a.

It is only recently that it has been realised that flue dust containing appreciable quantities of germanium could be obtained from most gas works in the U.K., and the last year has seen an intensive drive to find germanium in gas works that carbonise coal other than from the Northumberland and Durham coalfields, which were the ones originally discovered by Goldschmidt to contain appreciable quantities of germanium.

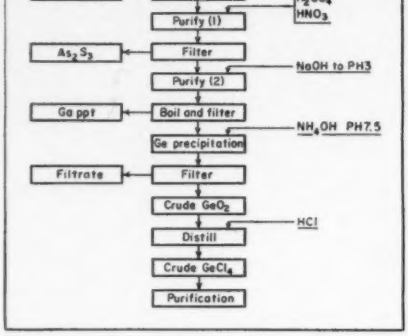
Morgan and Davies were the first to investigate the locations at which high-germanium-content flue dusts were deposited. They found that dust in the flues of producer plants generally contained more germanium than flue dust from the retorts in which the initial carbonisation of the coal took place. In general they found that the germanium content of the dust in the CO_2 flue and in the area around the waste heat boiler is greater than the germanium content of dust from the CO flue. This was attributed to the dilution of the dust in the CO flue by carbonaceous material, most of which, however, has been burnt by the time the dust and gases reach the region of the waste heat boiler and the CO_2 flue. An exception to this was found to be the South Metropolitan Gas Co. plant, where flue dust in the CO flue had a higher germanium content than that from around the waste heat boiler. The deposition of flue dust of high germanium content is highly localised and, in order to get a true picture of the flue dust in the plant, it is necessary to take samples from a number of parts of the flue system. The samples are best taken during the summer months when the annual overhaul is carried out, as investigators have found that samples taken from just inside the inspection points may not be representative of that area of the flue in general. Morgan and Davies found a spot in a flue system where a small sample of flue dust contained 1.6% gallium and 0.4% germanium. On analysing a sample weighing 3 tons, it was found that the average gallium and germanium contents were 0.30% and 0.23%, respectively.

It is comparatively difficult to ensure that, once having found a spot in a flue system at which flue dust with a good germanium content is deposited, similar flue dust will continue to be deposited at that particular point, as this depends on factors such as operating temperatures, type of coal carbonised and method of operation. For these reasons it is difficult to obtain large quantities of flue dust with a high germanium content. As the demand for germanium increases, more gas works are surveyed to discover if worthwhile quantities of enriched flue dust occur. By now, most large gas works in this country have been surveyed and their outputs are contributing to the supply of germanium metal in Britain.

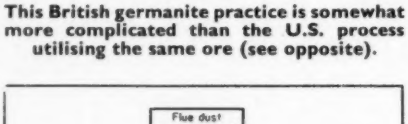
British Production Processes



This British germanite practice is somewhat more complicated than the U.S. process utilising the same ore (see opposite).



Only in the U.K. has commercial success been reported in the production of germanium from flue dust. American coals are being investigated for germanium.



Germanium tetrachloride is purified in this British process, hydrolysed to germanium dioxide, and the dioxide reduced. The American process for purifying the tetrachloride differs only slightly in that adiabatic columns are not used, and the copper refluxing step is omitted.

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Extraction process

The industrial extraction of germanium from germanite is based on the method devised by F. Sebba and W. Pugh.[4] The germanite was first ground to a particle size of -100 B.S.S. and added to a 50% solution of caustic soda, 1 cwt. of ore being added for every 60 lb. of caustic soda. The iron pot containing the mixture was stirred continuously with an iron paddle and the pasty mass was then cautiously heated and stirred until all the water had evaporated and a dry, porous, friable product obtained. The dry mass was leached with hot water in a stainless-steel pan and the leach liquors kept well stirred until the lumps of reaction product were completely disintegrated. After settling overnight, the clear orange liquid was siphoned off and the residue washed twice by decantation with hot water and, after filtering, washed again. The liquor and first wash were passed on to the next stage and subsequent washes used to treat the next batch of ore. In this way a strongly alkaline solution containing sodium thioarsenate, thiomolybdate and gallate was

obtained, together with other residues containing only small quantities of germanium. The alkaline solution was adjusted to *p*H 8 with 70% sulphuric acid, about 5% being treated with nitric acid in a stainless-steel pan and, when frothing ceased, heated to boiling point. Meanwhile, the remaining 95% of the solution was boiled in another pan and then allowed to flow slowly into the hot liquor in the first pan while the steam injection was continued. The nitric acid converted the germanium sulphide into soluble dioxide and precipitated a large proportion of the arsenic as sulphide, converting the remaining arsenic into arsenic acid without the action becoming uncontrollably violent. The crude germanium hydroxide was dissolved in sufficient hydrochloric acid to give, after the reaction was complete, a solution which was 6*N*. This solution was dissolved to recover crude germanium tetrachloride which was subsequently purified by distillation, and germanium dioxide was extracted.

Extraction from flue dust

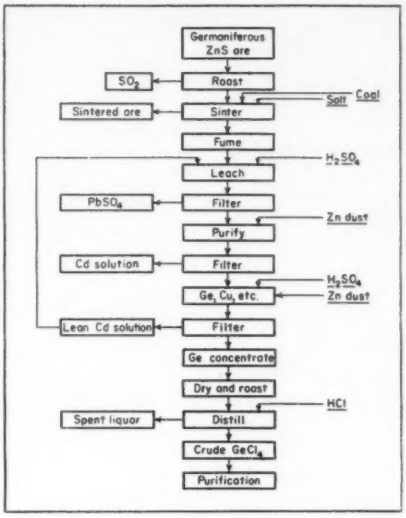
The production of germanium and gallium from flue dusts has been described by A. R. Powell, F. M. Lever and R. C. Walpole, of Johnson, Matthey Co. Ltd. After considering a number of chemical extraction processes, it was decided to investigate a metallurgical method of extraction, the initial step of which was the extraction of the germanium and gallium by smelting the dust with known collectors. Collectors suitable for germanium are iron, nickel and copper, and for gallium, generally copper. Flue dust was therefore smelted with soda and lime as fluxes for the silica and alumina, and with copper oxide and carbon to provide copper for the collection of the germanium and gallium. A copper-iron regulus was thus obtained which contained more than 90% of the germanium and 80% of the gallium. Dust high in sulphates was given a preliminary roasting to reduce the sulphur content to about 5%, which avoided the formation of excessive quantities of matte which, by taking up the copper, reduced the amount of gallium extracted into the regulus. The regulus generally contained 3 to 4% of germanium, and generally from 90 to 95% of the available germanium was extracted. Most of the germanium lost in the process is deposited in the flue of the furnace being used. The flue is therefore regularly cleaned and the flue dust returned for processing. To recover the rare elements from the regulus it was first converted into a solution of trichlorides, from which all the germanium, together with some of the arsenic, was recovered by distillation. The most convenient and economical method of doing this was to treat the regulus with a current of chlorine under a dilute solution of ferric chloride, which acted as a catalyst. The process is highly exothermic and could be carried out from cold without the application of external heat, the rate of the reaction being controlled by the rate of admission of chlorine into the system. The resulting chloride solution was siphoned or blown into a battery of *Pyrex* stills which were heated by electric mantles. The vapours passed through four standard *Pyrex* containers and the condensates flowed through a common pipeline to a 5-gal. *Pyrex* receiver. This was further connected through an empty carboy, which served as a trap, to a water-filled carboy in which non-condensed vapours of arsenic and germanium chlorides were collected and hydrolysed. Distillation was commenced by dropping slowly into the boiling solution sufficient concentrated sulphuric acid to liberate enough hydrochloric acid to render the solution in the still about 7*N* with respect to this acid. Distillation was continued until about 20 to 25% of the contents of the stills had been evaporated. Under these conditions the acidity of the condensate is such that it separated into two layers, the lower layer consisting of germanium tetrachloride containing up to some 20% of arsenic trichloride and the upper consisting of constant boiling hydrochloric acid containing large quantities of arsenic trichloride almost free from germanium. The crude germanium tetrachloride (boiling point, 84°C.) is separated from arsenic trichloride by careful fractional distillation. The purified germanium tetrachloride contained less than 20 p.p.m. of arsenic, which may be removed very slowly and incompletely by refractionation under the same conditions. The most satisfactory way of eliminating the residual arsenic is by prolonged refluxing of the liquid through a column packed with clean copper turnings. After refluxing for 12 to 15 hr., pure germanium containing less than 1 part arsenic in 10^7 germanium was obtained. The chloride was hydrolysed to dioxide and the precipitated germanium dioxide was washed free from hydrochloric acid and dried at 150°C. To produce germanium metal the oxide was placed in a pure graphite boat and heated in a tube furnace to a temperature of about 650°C. While the furnace was heating up to 650°C., pure hydrogen was passed through it until no further water was produced. At 650°C. the hydrogen was replaced with pure nitrogen and the temperature raised to 1,000°C. to melt the previously formed powder into an ingot. During the reduction process the temperature must be kept below 700°C. to avoid the loss of volatile germanium monoxide which was formed during the first stage of the reduction. Nitrogen or another inert gas should be used in the final melting operation to eliminate hydrogen from the metal, as an ingot moulded in a hydrogen atmosphere becomes porous on cooling, due to molten germanium absorbing an appreciable quantity of hydrogen which is liberated during solidification, producing minute blowholes throughout the ingot.

The method used in America for recovering germanium from residues of the zinc extraction process has not been disclosed in detail, although according to R. I. Jaffee, E. W. McMullen and B. W. Gonser[5] the main recovery step involves a distillation of germanium tetrachloride.

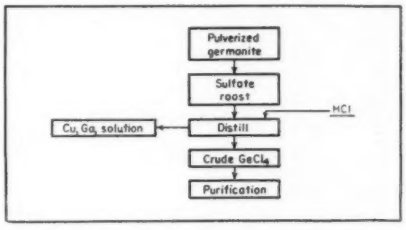
In the United States the leading producer of germanium is the Eagle-Picher Co., which has hitherto supplied germanium from residues from its zinc extraction process. The American Smelting & Refining Co. is working out an extraction procedure from the residues of its electrolytic zinc plant at Corpus Christi, Texas, and has announced that output may eventually rise to about 680 kg. a year. The American Zinc Lead & Smelting Co., of St. Louis, has a new zinc ore deposit from which some 2,500 kg. p.a. of germanium could be made available. The Anaconda Copper Mining Co. and various coal concerns are working on the possibility of extracting germanium, while investigations have also been made into recovering germanium from some Belgian Congo ores.

In this country, Johnson, Matthey Co. Ltd. are hoping to achieve an annual germanium output of about 350 kg. p.a.,

American Production Processes



This illustrates U.S. practice utilising zinc ores, and accounts for most of the germanium production there.



Some germanium is recovered commercially from germanite in this U.S. process, which differs somewhat from the British practice illustrated at the top of the opposite page.

using flue dust from producer plants as raw material. By this means it is hoped that the U.K. will soon not only be able to supply all home demands for germanium but also have some available for export.

The U.K. prices for germanium metal and germanium dioxide are about 5s. and 2s. 8d./gramme, respectively, for kilogramme lots. In the United States the prices of metal and dioxide are 75 cents and 31¼ cents/g.

Uses of germanium

The demand for germanium has risen sharply since the war, mainly on account of its use in the manufacture of new types of diodes and triodes. Silicon diodes were developed during the war to rectify very high frequency voltages, but they could only handle comparatively small voltages (generally less than 10 v.) and, in any case, their performance tended to deteriorate when used at frequencies below about 50 megacycles. It was found that a germanium rectifier could handle up to 100 v. and still maintain a high electrical resistance, which is one of the criteria for satisfactory rectification. No filament heaters are required with germanium diodes and so, not only is heater current saved, but the electrical capacity between the filament and cathode is eliminated. Germanium diode rectifiers have been found suitable for practically all the purposes for which thermionic diodes are now used. In particular, germanium diodes can be used in the discriminator circuit of frequency modulated recivers and as final detectors in radar and television video and sound receivers. Other obvious applications of germanium diodes are in meters which measure very high frequency currents and voltages and in modulators for telephone equipment.

The amount of germanium metal used in each diode is comparatively small and is generally about 10 mg. The diode consists of an electrode (the familiar cat's whisker of early radio days) held against the surface of the piece of germanium, generally by a thin layer of plastic cement. As the whole diode is encased in a strong, waterproof case it is more robust than the glass-envelope thermionic types and, provided that a germanium diode is not overloaded or otherwise mishandled, it should have a very long lifetime, certainly much more than the 1,000 to 10,000 hr. of the thermionic valve.

In 1948 the Bell Telephone Laboratories, New York, announced the introduction of a germanium crystal triode, now called a transistor, which contained two electrodes placed a hair's breadth apart on the surface of a piece of germanium. By applying suitable D.C. voltages to the electrodes the transistor can be made to act like a triode valve and under suitable operating conditions can give both voltage and current amplification. The original types suffered from a number of weaknesses such as their fragility, restricted frequency range and their comparatively high noise figure. The latter defect meant that if they were used in an ordinary broadcast receiver the background noise would be much higher than when using thermionic valves.

Since Bell Telephone Laboratories first announced the discovery of the type A transistor, as it is now called, several other forms have been developed and it is likely that transistors with better frequency response, lower noise figure and capable of handling larger currents will become available. Already the performance of the Junction-type transistor has been found to approach that of some thermionic valves.

Transistors have much the same advantages as germanium diodes in that their small size, ability to withstand rough treatment and absence of filament supplies make them particularly attractive for specialised uses such as the manufacture of airborne radio and radar equipment, electronic computors and hearing aids.

Production of a number of transistors each having the same performance is a comparatively difficult operation to achieve and a very careful control of germanium purity is required. Impurities should not exceed 1 part in 10^7 and those that are present should be distributed homogeneously throughout the metal.

For certain transistors, germanium in single crystal form is used and it has also been found necessary to develop techniques such as heat and electrical forming for controlling the production of semiconductor properties. It has recently been announced that the U.S. Signal Corps has placed $5.4 million worth of contracts for the development of germanium diode and triode production equipment. In the States these contracts are being shared between four leading companies: General Electric, Radio Corporation of America, Silvania Electric Products and Raytheon Manufacturing Co. It is stated that an important part of the machinery development programme will be the mechanisation of operations to produce germanium metal. In this country most leading radio and television companies are investigating the manufacture of transistors and in 1951 the General Electric Co. demonstrated a domestic radio receiver using transistors in place of the conventional thermionic valves. It is doubtful, however, if many transistors will be used, at any rate initially, for domestic radio receivers.

The use of germanium in the manufacture of diodes and triodes has resulted in increased supplies of germanium dioxide and germanium metal being available in this country. This in its turn has increased interest generally in germanium and since the war a number of different uses, outside the electronic field, have been announced. Some experiments have been carried out in which germanium has been substituted for silicon in certain alloy steels. By this means the high-frequency characteristic of the steel is improved and the resulting metal may be used in the manufacture of laminations for transformers. This type of alloy may also have some application in the manufacture of high-frequency conductors.

A considerable amount of academic work is also being carried out on germanium alloys. H. J. Axon and W. Hume Rothery have published a paper entitled 'The Lattice Spacing of Different Elements in Aluminium,'[6] in which a description is given of the measurements of the lattice spacings of a solid solution of a number of elements, including germanium, in aluminium. It was found that the aluminium lattice expanded when the solutes were magnesium and germanium, and contracted when they were lithium, silicon, copper and zinc. No change of lattice spacing occurred when silver was dissolved in aluminium. The crystal structure of the solute at 100% concentration was compared with that of the same solutes dissolved in univalent metallic solutions such as copper, silver and gold, and a divalent solution such as magnesium. Axon has also carried out work[7] in connection with the systematisation of certain binary metallic equilibrium systems. He found that two main classes of systems were formed. In the first class a region of liquid immiscibility occurred, while in the second class a eutectic point was found.

R. I. Jaffee describes[8] an investigation into the germanium-gold alloy system and the discovery of the eutectic point at which the composition of the alloy was 12% germanium and the temperature 356°C. Although it has been found impossible to verify if similar work is being carried out in the U.K., it has been said that dentists are beginning to make use of the germanium-gold alloy, as the low melting point of the eutectic alloy and the property of expansion on freezing makes it very suitable for the manufacture of dental casts.

Work is thought to be proceeding on a number of germanium alloys such as germanium/zinc and germanium/magnesium alloys, but as these are generally brittle they will probably have only a narrow and specialised application. A considerable amount of work is being carried out on germanium alloys, but it is too early yet to decide which will eventually be commercially important.

Experiments are being performed in this country on the use of a germanium compound for coating the inside surface of fluorescent lamps. The spectrum obtained from a fluorescent lamp using this coating is claimed to be nearer to that of white light than that from other types of fluorescent lamps. The germanium for this work is generally used in the form of manganese

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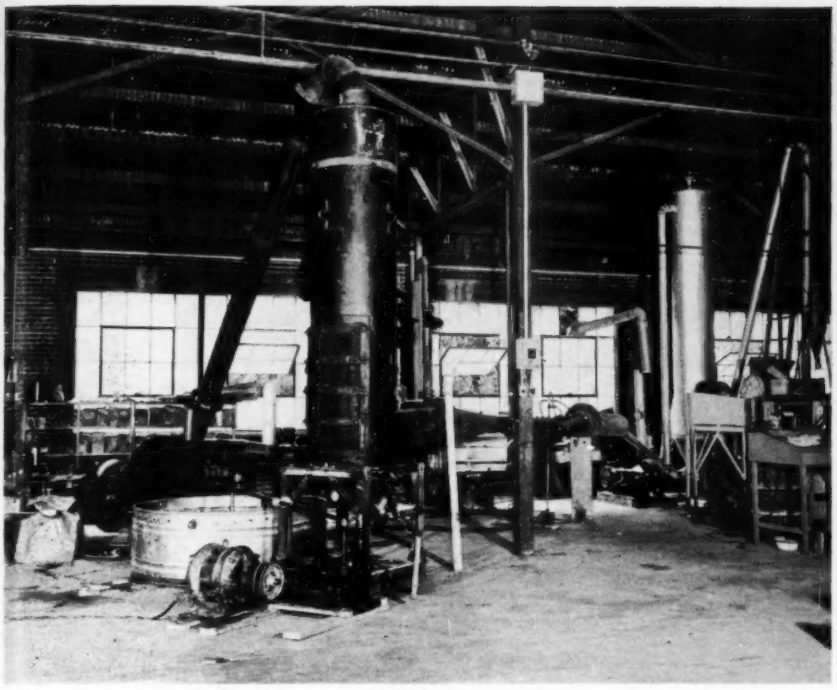

Elaborate fume and vapour recovery devices are essential in studying the potential utilisation of new germanium sources. The pilot plant shown is smelting germaniferous ores.

germanite powder and must be free from heavy metal contaminants such as nickel, cobalt and iron and from sulphide and chloride radicals. The germanium should not contain more than 5 parts of impurity to 10^5 parts of germanium (this is an appreciably higher impurity content than the germanium used in the manufacture of germanium diodes and transistors). At its present price of 5s. g., germanium is too expensive to use on a wide scale for this application and it seems that the price would have to fall very substantially before it would be commercially worth while, although the higher impurity content would probably permit a rather lower price.

Germanium is optically transparent to radiation, having wavelengths between 2.5 and 15 microns, part of the infra-red spectrum. A number of experiments have accordingly been made in this country and the United States on the use of germanium in the manufacture of lenses and prisms for use in infra-red frequency work. R. G. Trenting[9] describes an acromatic doublet made from lenses of silicon and germanium. As impurities in the germanium form opaque spots in the lens or prism, it is necessary to use the very purest type of germanium metal, probably metal with an impurity content of not less than 1 part impurity to 10^7 parts germanium. With germanium at its present very high price, it is doubtful, however, if numbers of prisms weighing, say, 70 g., will be manufactured unless the price of germanium falls considerably.

Fluxmeters for measuring the intensity of magnetic fields have been manufactured from small pieces of germanium. This application makes use of the ' Hall effect,' in which a magnetic field is made to produce a voltage proportional to the magnetic intensity. A piece of germanium, weighing perhaps 60 mg., is used in the manufacture of this type of fluxmeter; the detecting head of the instrument may therefore be inserted into very small volumes, such as the field gap in a moving coil pick-up. There are practical difficulties in welding the two sets of contacts on to the metal, but, when these have been overcome, a simple and robust instrument results. One of the first uses for this type of fluxmeter was in the synchronisation of the peak of an alternating magnetic field in a synchratron with the time of injection of elementary particles. As the instrument is so simple and robust, it is likely that it will eventually be widely used in school laboratories.

Alpha particle counters can be manufactured from germanium, as their energy is usually sufficient to ionise the atoms of germanium by removing an orbital electron. The Atomic Energy Research Establishment have already demonstrated this device and shown that an ordinary germanium diode with a small voltage potential across it can be used to show the effect. Suitable electronic equipment is, of course, necessary to record the scintillations and at present this use of germanium is almost entirely academic.

In the last few years experiments have been carried out into the properties of glasses containing germanium. Various experiments have been carried out to see if germanium glasses are suitable for infra-red frequency work, while optical glasses having a refractive index greater than 1.7 have also been made. Although the properties that germanium gives to certain glasses are useful, it is unlikely that they will be employed on a wide scale except for very specialised uses.

B.T.H. have described[10] a method of applying a thin film of germanium to a hot surface. Finely divided germanium dioxide or germanium oxide is introduced into the reducing portion of a low-velocity gas flame and the tip of the flame directed on to the surface to be coated. The temperature of the gas flame in contact with the germanium dioxide should be near to the melting point of germanium (959°C.). The germanium dioxide is reduced to the metal by the action of the flame and the particles of molten germanium metal are carried by the flame and deposited on the solid surface, where the germanium solidifies and forms an even film. It was found that the maximum thickness of the film was proportional to the thermal conductivity of the surface. By examining the film by an electron diffraction method, the size of the primary crystals was estimated to be about 2,000Å.

It can therefore be seen that the major use of germanium is in the semi-conductor field for the manufacture of diodes and transistors. A number of experiments, both here and abroad, are being carried out into the use of other semi-conducting materials such as silicon and lead sulphide. So far, however, it has been found very difficult to make a very pure form of silicon, as the melting point of silicon is much higher than that of germanium, and contamination from crucibles in which the metal is held generally results. Lead sulphide is attractive as a semi-conductor material, but suffers from the disadvantage of being rather fragile, and it is therefore likely that, unless its properties are outstandingly better than those of germanium, it will not be used very much outside laboratories.

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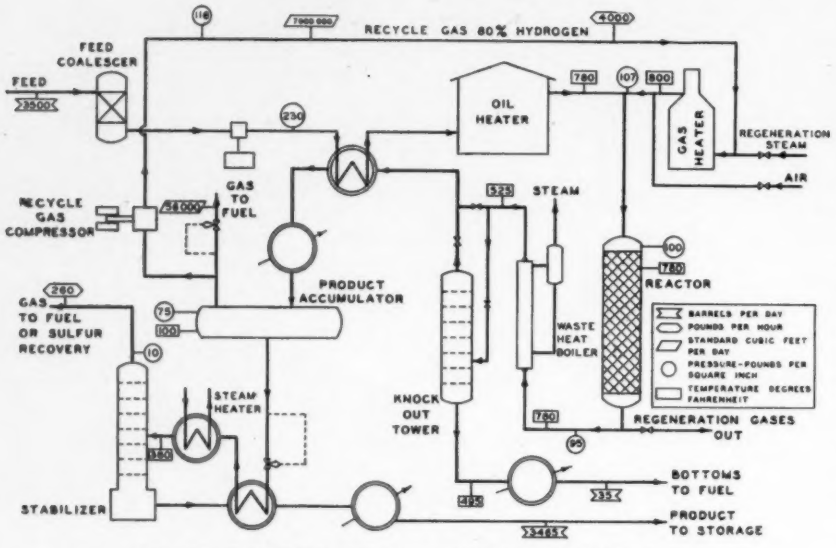
Catalytic Desulphurisation

New 'Autofining' process suits wide range of petroleum feedstocks

A new catalytic desulphurisation process named 'Autofining' has been developed by the Anglo-Iranian Oil Co. for the treatment of petroleum feedstocks. The process has been fully proved in extended operations in a 350 B.P.D. pilot plant, and the first commercial-size unit of 3,500 B.P.S.D. input capacity at National Oil Refineries Ltd., Llandarcy, South Wales, has been in operation for several months.



Flowsheet of 3,500 barrels day autofining unit at Llandarcy

THE *Autofining* process operates at pressures of 50 to 300 lb. gauge and at temperatures of 700 to 800°F. and makes use of a very stable sulphur-resistant catalyst which has a long life. The catalyst is regenerated periodically, the on-stream period varying with the boiling range of the feedstock. A small amount of gas, consisting mainly of hydrogen, is produced in the process and this is recycled. No hydrogen manufacturing facilities are required.

Constant-pressure operation is normally employed with the lighter feedstocks. A small excess gas make is vented from the system to keep the pressure constant. It is desirable, however, when maximum sulphur removal is required, to operate the unit under equilibrium-pressure conditions. Under these conditions full use is made of the hydrogen produced and no excess gas, except that dissolved in the liquid product, is removed from the system.

A wide variety of feedstocks can be effectively desulphurised, ranging from natural and straight-run gasolines through naphthas and kerosines to gas oils and light diesel fuels. Sulphur removed from the feedstocks is largely converted into hydrogen sulphide. The products are of good colour and odour and are very stable. They need no subsequent refining treatment. The yields of liquid products obtained are 99% plus by weight or about 100% by volume.

The process is covered by patents or patent applications in all major countries.

Operations at Grangemouth refinery

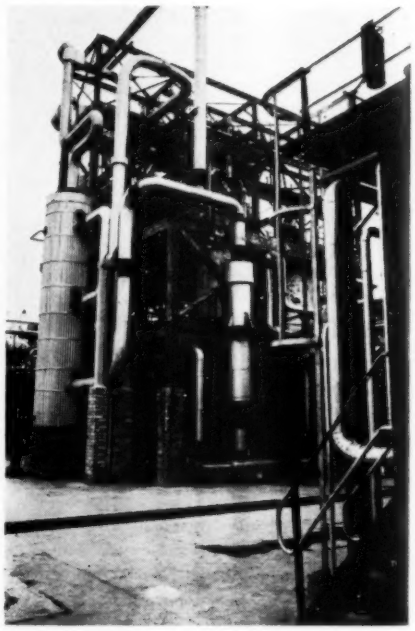
At Scottish Oils' Grangemouth refinery, Scotland, a 150-barrel day pilot plant was commissioned in 1948 and this was subsequently enlarged to 350 barrels day in 1949. Since May 1950 the unit has been used as a production unit to desulphurise a kerosine sulphur dioxide extract which is used as a high-octane blending component in power kerosines (tractor vaporising oils). The unit operates at 3 v. v. hr., about 100 p.s.i.g. pressure, 2,000 s.c.f. b. recycle rate and 780°F. temperature; the on-stream period between regeneration is about 200 hr. The catalyst charge has, so far, been in use for 16,000 hr., during which period it has been regenerated about 100 times. Typical results on this unit are:

	Feedstock	*Product*
Specific gravity, 60 F./60 F.	0.870	0.867
Gravity, °A.P.I.	31.1	31.7
I.B.P., °F.	327	311
10% vol. at °F.	347	347
50% " " "	388	383
90% " " "	486	483
F.B.P., °F.	563	567
Total sulphur, % wt.	1.04	0.45
Sulphur removal, %	—	56.8
Octane No. (MM) clear	76	77

Operations at Llandarcy refinery

A 3,500-barrels/day unit operating on a naphtha for petroleum spirits (white spirit) and power kerosine (tractor vaporising oils) in blocked operation was commissioned in July 1952. A simplified flow sheet of the unit is given on this page and the photographs give details of the various items of the equipment.

In this unit the feedstock and the recycle gases are heated in separate heaters and pass downwards through the reactor. The hot vapours from the reactor pass through a waste heat boiler, where steam is generated; after heat exchange and cooling, the products flow to the separator from which the recycle compressor takes suction. If it is desired to meet a colour specification with products such as power kerosines, the products leaving the waste heat boiler pass into the knock-out tower where about 1 to 2% volume of heavy ends are removed. Any excess gas make is passed to fuel from the product separator. Liquid product from the separator passes to a flash tower stabiliser to remove the hydrogen sulphide and other gases which results in flash point rectification. The stabiliser product is normally free of hydrogen sulphide, but a light caustic soda wash is given to guard



Reactor (*left*) **with product coolers** (*top right*)

against poor stabiliser operation.

The design conditions for this unit on either feedstock are:

Pressure	100 p.s.i.g.
Reactor temperature ..	780° F.
Space velocity	3.0 v./v./hr.
Gas recycle rate ..	2,000 s.c.f./b.

and the following data are typical of the results obtained when operating under these conditions:

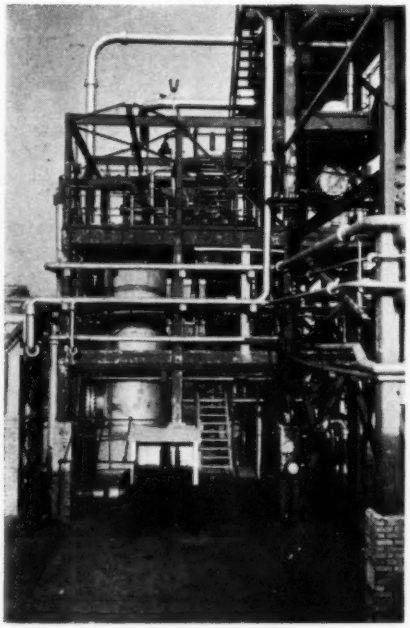
Power Kerosine (Tractor Vaporising Oil)

	Feedstock	*Product*
Specific gravity, 60° F./60° F.	0.824	0.820
Gravity, °A.P.I. ..	40.2	41.1
I.B.P., °F.	309	298
10% vol. at °F. ..	336	329
50% " " " ..	383	381
90% " " " ..	460	453
F.B.P., °F.	511	496
Sulphur, % wt. ..	0.645	0.106
Sulphur removal, % ..	—	83.6
Colour Saybolt ..	+10	+21
Corrosion (Cu strip at 212° F.)	Pass	Pass
Octane No. (MM) clear	43.5	47.0

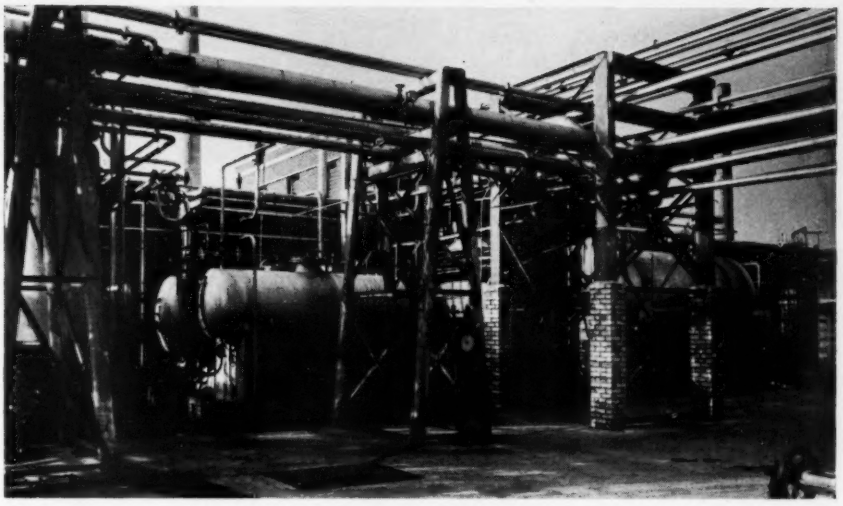
Approximately 1.5% volume bottoms was removed in the knock-out tower.

Naphtha for Petroleum Spirit (White Spirit) Production

	Feedstock	*Product*
Specific gravity, 60° F./60° F.	0.780	0.780
Gravity, °A.P.I. ..	49.9	49.9
I.B.P., °F.	302	293
10% vol. at °F. ..	318	318
50% " " " ..	338	338
90% " " " ..	374	372
F.B.P.	408	412
Sulphur, % wt. ..	0.143	0.003
Sulphur removal ..	—	97.9



Knock-out column (*left*) **and waste-heat boiler** (*centre*) **with reactor** (*right background*)



Coalescers (*extreme left*), **caustic soda vessels** (*left centre*) **and separator** (*right*) **with control and pump house** (*background*)

Utilities

The consumptions of utilities in the 3,500-b.p.s.d. commercial unit at Llandarcy when operating under full design conditions are:

150 lb. steam ..	6,600 lb./hr. (net)
Cooling water ..	30,000 Imp. gal./hr.
Fuel	25,000,000 B.Th.U./hr.
Electric power ..	10 kw.

In this plant, the recycle gas compressor is driven by the steam generated in the waste heat boiler.

Investment costs

The 3,500-b.p.s.d. commercial plant had a total erected cost within battery limits of approximately £250,000. Of this total cost, materials, including common building materials, draughting, engineering, procurement and contractors' overhead and profit, amounted to £196,000.

New Books

Organic compounds of phosphorus

Although the organic chemistry of phosphorus compounds is in its second century, no comprehensive coverage of the subject was attempted until 1938, when a Russian volume appeared, which was not, however, sufficiently detailed to enable the reader to locate substances individually.

Now, what is claimed to be a complete modern coverage* of all known organophosphorus compounds with their physical and chemical properties has appeared. It includes synthetic methods of preparation for all known classes of organophosphorus compounds, including some information which English-speaking chemists either could not formerly obtain, or could only obtain from many scattered sources.

All chapters follow the same general pattern of brief introductory statement about the compounds with which they deal and details of synthetic methods applicable to them. They cover phosphines and related compounds; halophosphines; halophosphine halides and phosphonyl halides; quaternary phosphonium compounds; tertiary phosphine oxides, sulphides and selenides; phosphinous, phosphonous and phosphonic acids, their sulphur analogues and esters; phosphites and thiophosphites; phosphates, halophosphates and thio analogues; compounds with phosphorus to nitrogen bonds; quasi-phosphonium compounds; and derivatives of anhydro acids.

**Organophosphorus Compounds*, by G. M. Kosolapoff. Chapman & Hall. Pp. 376. 60s. net.

Practical physical chemistry

This is a review of physical chemistry, written for the most part at post-matriculation or intermediate level and with the stress upon calculation.* It is especially addressed to those whose mathematics are below average and explanations of certain calculations are given in considerable detail. The book will probably be useful, even when it is not prescribed, to those students who find the subject difficult.

The author is an industrial chemist, who has related principles where possible to everyday practical processes and it is hoped that certain sections will remind students of the relevance of their studies to recent scientific developments.

Seventeen chapters deal with gases, eudiometry, liquids, solids, solutions, the distribution law, osmotic pressure, vapour pressure of solutions, boiling point and freezing point, mass action, velocity and order of reactions, the phase rule, thermochemistry, electrolysis and electrolytes and the structure of the atom.

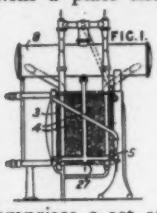
**Physical Chemistry Made Plain*, by J. H. Mandleberg. Cleaver-Hume Press, 1952. Pp. 287, including index. 15s. net.

Chemical Engineering Invention

MONTHLY SUMMARY OF BRITISH PATENT CLAIMS

Evaporators

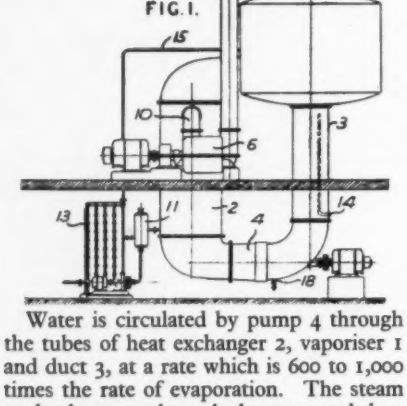
An evaporator for whey and similar liquids comprises an evaporation chamber 8, Fig. 1, under reduced pressure, communicating with a plate heater 3, 4, 5.



The latter comprises a set of plates 4 in which the whey is heated and circulated to and from the evaporation chamber, and a set of plates 3 in which fresh whey is preheated by condensate from the set of plates 5 in which the evaporated vapours are condensed. The evaporated product is withdrawn batchwise or continuously from pipe 27.—642,166, *Aktiebolaget Separator.*

Vapour-compression distillation

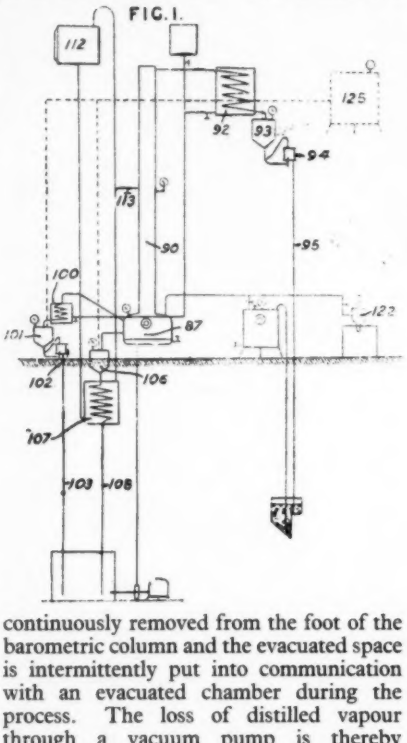
In the distillation of water by continuously circulating it through a heat exchanger and a vaporiser, compressing the vapour from the vaporiser and passing the compressed vapour to the heat exchanger, the water is treated for the precipitation of impurities as a slurry. The rate of circulation of the water is maintained several hundred times greater than the rate of evaporation.



Water is circulated by pump 4 through the tubes of heat exchanger 2, vaporiser 1 and duct 3, at a rate which is 600 to 1,000 times the rate of evaporation. The steam evolved passes through dome 17 and duct 9 to turbo-compressor 6 which operates at a compression ratio of 1.1 to 1.4. The compressed steam is supplied to heat exchanger 2 through duct 10. Electrolysis of the water in duct 3 is effected by passing an electric current between aluminium electrode 14 and the walls of the circulating system. This causes the impurities to form, together with aluminium hydroxide, a loose slurry which is continuously withdrawn by draining off a small fraction of the circulating water through duct 18. Raw water, preheated in heat exchanger 13 by the steam condensate (*i.e.* distilled water) from heat exchanger 2, is supplied to vaporiser 1 through line 15. With an effective temperature difference of about 5°C., across heat exchanger 2, the increase in temperature of the water during its passage through the heat exchanger is 0.5 to 1°C. Thus only a small hydrostatic head is required to prevent ebullition in the tubes. The plant is adequately heat-insulated. The gases disengaged by condensation of steam in the heat exchanger 2 are withdrawn from vessel 11. If non-corroding steel tubes are used for the heat exchanger 2, the thickness of their walls should not exceed 1.25 mm.—641,429, *R. Andersen.*

Vacuum distillation; alcoholic liquors

Vacuum distillation is carried out by continuously distilling a liquid from a boiler through a fractionating column into the closed evacuated space at the top of a barometric column. The distillate is

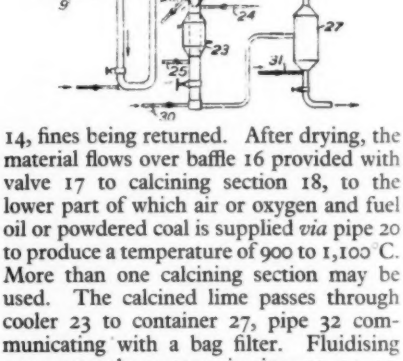


continuously removed from the foot of the barometric column and the evacuated space is intermittently put into communication with an evacuated chamber during the process. The loss of distilled vapour through a vacuum pump is thereby eliminated. The exhaustion of aqueous-alcoholic liquids to an ascertainable degree of alcoholic-exhaustion is referred to.

The distilland is supplied from tank 112 *via* preheater 107 to ring-packed fractionating column 90, which it enters through valve 113. The column is connected at the base to boiler 87 and at the head to condenser 92, container 93, test-piece 94 and barometric column 95. Prior to the admission of distilland to the column, the apparatus is evacuated by pump 122, but when this has been completed, all communication between the apparatus and a vacuum pump is cut off. During the distillation, the container 93 is periodically put into and out of communication with an evacuated chamber (not shown) by device 125. The hot distillation residues leave the boiler and pass through container 106, preheater 107 and hydraulic column 108. The vapour-space of the boiler is connected to condenser 100, container 101, test-piece 102 and hydraulic column 103. In the distillation of alcoholic liquids, test-piece 102 indicates, by calibrated adjusting gear, whether distillation is being performed to the desired exhaustion. If this is not the case, the rate of boiling is increased or the rate of feed is diminished. Containers 101 and 106 are both connected to the boosting device 125. The plant may be constructed of iron, galvanised copper or aluminium.—642,127, *R. F. Gill.*

Burning lime

Limestone is calcined whilst in the form of a dense fluidised bed. As shown, the material from crusher 1 and pulveriser 3 is fed to a container 5 wherein it is aerated by flue gas, air, etc., from pipe 9. The fluidised powder passes to the upper part of reactor 10 wherein it is dried by hot gases from the lower sections passing through grid *G*. The gases pass through cyclone separator 12 and waste-heat boiler



14, fines being returned. After drying, the material flows over baffle 16 provided with valve 17 to calcining section 18, to the lower part of which air or oxygen and fuel oil or powdered coal is supplied *via* pipe 20 to produce a temperature of 900 to 1,100°C. More than one calcining section may be used. The calcined lime passes through cooler 23 to container 27, pipe 32 communicating with a bag filter. Fluidising gases enter the system *via* pipes 19, 24, 25, 30, 31. In a modification, part of the calcined lime from the exit is returned to the calcining section *via* a heat exchanger.—642,036, *Standard Oil Development Co.*

Plant and Equipment

Reflux dividers for batch stills

The *Spotton* reflux divider is described by its makers, Metal Propellers Ltd., as a simple and efficient device for controlling the reflux ratio for batch stills. It is claimed that it will permit rapid setting of selected ratios and their reproduction when ever required. Flow through two rectangular ports is controlled by a single lever operating on a calibrated scale. The advantage of the rectangular ports is that their total width is always constant but the relative width is variable, thus giving a continuously variable reflux ratio over a wide range. This device is produced in three sizes: No. 1 size with a maximum range of input of 10 to 150 gal./hr., No. 2 allowing 100 to 1,000 gal./hr. and No. 3 allowing 500 to 4,000 gal./hr. All models can be supplied in stainless steel, *Monel*, nickel or copper. A No. 2 model was displayed at the B.I.F. at Birmingham this year, with a sight glass attachment which can be fitted to the product outlet.

'Spotton' No. 2 reflux divider and sight glass attachment

Vibration tester

Chemical plant is rapidly increasing in size and there is a corresponding rise in the reaction pressures involved. This trend has increased not only the difficulties of design but also of construction and maintenance problems. With large capital sums involved, it is strongly advisable to check plant periodically. For this work, non-destructive tests are now becoming popular in which portable electronic instruments are used to check the soundness of plant while it is in operation. Typical is the ultrasonic thickness gauge which will measure wall thickness when only one side is available (CHEMICAL & PROCESS ENGINEERING, October 1952, p. 561). Wear and erosion are thus readily detected. Another useful instrument is the improved Dawe type vibration meter. This meter shows the amplitude of vibration in any machine, structure or other solid matter immediately a probe is placed in contact with it.

The meter is normally suspended from the operator's shoulder by a strap so that he has both hands free to climb about the superstructure of the plant. Once in position, the probe is held or clamped against the structure while the reading is being taken.

The usual procedure is to take vibration readings at salient points all over the plant before it is put into operation. Any large vibrations are treated by strengthening the structure. Checks are then made at frequent intervals. Any rapid increase in vibration indicates a point at which wear is taking place and which should be carefully watched. Once the normal operation of the plant is known, it is advisable to take routine checks at points of rapid wear, say every week. This is supplemented by thorough measurements at less frequent intervals to check the condition of the whole structure. Fuller details of this instrument are available from Dawe Instruments Ltd.

PTFE coating for feed-through rollers

The Edison Swan Electric Co. Ltd. have introduced a process whereby rollers and other cylindrical parts can be coated with polytetrafluoroethylene (PTFE). This process should be of particular interest to food manufacturers and others using continuous feed-through rollers as, it is claimed, the properties of PTFE are such that sticking and contamination are virtually impossible.

The manufacturers state that PTFE is a new resin-like substance unique among organic compounds in its chemical inertness and in its toughness over a wide range of temperatures. It withstands the attack of all materials except alkali metals; it can be boiled in alkali hydroxides, hydrofluoric acid, fuming nitric acid or aqua regia with no change in weight or properties. It also resists the attack of all high-boiling solvents and other organic compounds.

The material has zero water absorption, high impact strength and form stability, a far greater resistance to chemicals than either gold or platinum and retains its strength at temperatures ranging from -100°C. to $+288^{\circ}$C.

Available also in rod, sheet and tape form, PTFE is already being widely used in the chemical and allied industries for gaskets, joints, valve seats, etc.

Potentiometer, designed for easy portability and fitted into carrying case

Portable potentiometer

A new portable potentiometer by W. G. Pye & Co. incorporates many of the features of their precision-grade laboratory instruments in a carrying case measuring 12 in. × 10 in. × 7 in. and weighing 12 lb. The potentiometer circuit comprises a main dial switch and a circular slide wire. On the fundamental range, the main dial gives steps of 0.1 v. and the slide wire gives continuous sub-division of one step. The scales carried by these two components are visible behind two adjacent windows, and the answer is given by reading straight across the windows. Three ranges are provided: 0 to 1.8 v., 0 to 180 mv. and 0 to 18 mv. The accuracy of the instrument is $\pm 0.1\%$ or ± 1 slide-wire division, which ever is the larger.

To obviate the necessity of balancing the potentiometer against the standard cell by altering the main and slide-wire dials, a special circuit is incorporated into the instrument. All that is necessary is that the push-switch marked *standardise* be pressed intermittently and the galvanometer brought to zero by adjustment of the *coarse* and *fine* controls. This device is particularly useful where speed is important and it allows the potentiometer to be checked for standardisation with the minimum of effort during a series of measurements. A miniature standard cell is built into the instrument and is specially chosen to give long trouble-free life. A galvanometer of the taut-suspension type is incorporated. The scale has a centre zero only, as the galvanometer is used as a null detector in the potentiometer. Zero adjustment is easily effected by the rotation of the large plastic knob which protrudes above the panel. The galvanometer is automatically protected when the lid of the instrument is closed. It costs £50.

For further details of plant and equipment please use the coupon on p. 188

World News

GREAT BRITAIN

Roscdowns to make Merz solvent extraction plants

Rose, Downs & Thompson Ltd. have concluded an agreement with Merz Patents Ltd. for the manufacture and sale under sole licence of the whole range of Merz solvent extraction plants, both batch and continuous. The full-time services of Mr. Victor Merz have been retained by the new licensees and he has joined their organisation at Hull.

The company are now able to offer complete ranges of plants for the treatment of oilseeds or other oil-bearing materials by pressing, expelling or solvent extraction, or by a combination of any of these processes.

In addition, they are able to offer complete plants for the after-processing of the oils produced, either for edible or industrial purposes.

Utilisation of low-grade ores

A mission from the Organisation for European Economic Co-operation visited the U.K. in April to gather facts on research into the exploitation of low-grade ores. The party spent five days in visiting laboratories and mines and held informal consultations with British experts. The mission is also visiting France, Germany, Italy and Sweden.

It is hoped that, as a result of the mission, closer co-operation between organisations interested in mineral dressing will come about and methods will be developed of mining and dressing low-grade ores which have as yet been uneconomic to mine.

The mission includes experts from Austria, Belgium, France, Germany, Greece, Italy, Norway, Sweden, Turkey and the U.S. British representatives are Prof. J. A. S. Ritson, and Dr. W. Davies, head of the ore-dressing laboratory of the United Steel Companies Ltd. The secretary of the mission is Mr. J. Sandor, of the Department of Scientific and Industrial Research.

Among the establishments visited by the party were the mineral-dressing laboratories at Harwell, the research laboratories of the General Electric Co., the Bessemer laboratories of the Royal School of Mines and mines at Plymouth, St. Austell and Pendeen.

At the end of the tour in the U.K. the mission met for informal discussions at the headquarters of the D.S.I.R.

Chemical stoneware production

Aerox Ltd., of Glasgow and Crawley, have entered into association with Transport & Chemical Equipment Ltd. for the production of *Petrox* chemical stoneware. This takes two forms, *Petrox A*, a white felspathic porcellaneous body fired to a high temperature and completely vitrified, claimed to have considerable mechanical strength and resistance to thermal shock, and *Petrox B*, a dense grey vitrified non-porous body which can be supplied glazed or unglazed and which is of especial value where unusually high resistance to thermal shock is required.

Standard production items include tanks, acid filter funnels, evaporating basins, armoured pressure valves, acid jugs, flat trays, storage coppers, Buchner funnels, vacuum filters, ball mills and plug cocks.

Babcock & Wilcox retirement

Mr. W. R. Clark Lewis, M.Inst.T., has retired from the position of traffic manager to Babcock & Wilcox Ltd., after 45 years' service.

Mr. Lewis first joined Babcock & Wilcox Ltd., in the erecting department, in 1907, and transferred to traffic work in the invoice department in 1911, eventually taking charge of the traffic department on its inception in 1933.

Mr. Lewis is succeeded as traffic manager (London) by Mr. K. J. McBryde, who joined Babcock & Wilcox in 1950. He was previously on the staff of the Commercial Superintendent, British Railways (Western Region), where he had obtained some 20 years' experience in railway traffic problems.

New Monsanto plants

The final stages of the new biological effluent treatment plant at the Ruabon factory of Monsanto Chemicals Ltd. have been put to work. Regarded as the latest in technology in the field, the plant is proving satisfactory in every way. This information is given in the chairman's statement for 1952. Also commissioned during the year at Ruabon were: a fifth unit for the production of phthalic anhydride; increased capacity for the production of synthetic phenol, aspirin, phenacetin and other related pharmaceuticals; necessary additional warehouses; and 'the first stage of a major pilot-plant unit for interim production of promising new products.' A major project for the manufacture of *Syton*, used in the treatment of textiles, was completed and put into commercial-scale operation. At the Newport factory a new unit for the production of *Stymer*, another product used in textiles, was completed, as was also a unit for producing organic silicates.

At the company's new research laboratories at Fulmer Hall, Bucks, further facilities have been brought into use for research on new applications for plastics, adhesives, rubber chemicals, fungicides and agricultural and textile chemicals. In addition, engineer testing equipment for the development of chemical additives for treating lubricating oils were brought into full operation. The staff at Fulmer Hall numbers about 100, of whom 70 are scientific and technical graduates.

Production of the synthetic polyelectrolyte resin soil conditioner, *Krilium*, commenced during the latter part of the year. In addition to its uses as a soil conditioner, it has other applications, including erosion control.

Financial results. Net profit, after tax, was £338,485 in 1952, compared with £939,233 in 1951. The figures for 1949 and 1950 were £318,085 and £582,308, respectively. The chairman stated that during 1952 there was an emphatic shift from a seller's to a buyer's market and profits suffered because of lower selling prices forced by intensive competition, especially in export markets, and continued increases in raw material and labour costs. Nevertheless, the total value of goods sold was maintained at only 3% below the 1951 level, and the tonnage of goods sold showed an increase of approximately 10%. Exports were increased to 39% of total sales, compared with 33% in 1951.

Boiler research

A new cyclone furnace boiler built by Babcock & Wilcox Ltd. to afford full-scale facilities for experimental work on cyclone operation is nearing completion and expected to be in operation shortly. It has an evaporative capacity of 60,000 lb. hr. and, like the other three boilers at present available for research work, will carry a portion of the steam load of the Renfrew works. It is expected to provide a great deal of valuable information on cyclone and boiler operation and performance, under a wide range of steaming conditions, with a variety of British and foreign coals.

Another boiler at Renfrew, a new integral furnace unit, has been specially designed to facilitate experimental work on fuel mills and has comprehensive arrangements for fuel weighing and facilities for rapid changeover from its normal 'E' mill firing to any other type of mill, either on direct firing or on the bin-and-feeder system. Fired by pulverised fuel, it has a capacity of 60,000 lb. hr.

Institute of Fuel luncheon

The annual luncheon of the Institute of Fuel, held in London recently, was attended by some 600 members and guests. The Melchett Medal for 1953 was presented to Dr. Harold Hartley, C.B.E., for his work on domestic fuel appliances. The principal guest was the Minister of Fuel, who said that we are facing competition from other states that have a much greater fuel potential than ourselves. In the U.S. the *per capita* fuel consumption is nearly twice that of ours, while in Russia it is expected that coal production in five years' time will be nearly double that in the U.K.

Dr. G. E. Foxwell, president of the Institute, spoke of the need for training more fuel technologists, and Mr. H. F. H. Jones, M.B.E., deputy chairman of the Gas Council, reviewed the fuel policy of the Council.

Power-Gas appointment

Mr. R. W. Rutherford, M.I.Chem.E., has just been appointed deputy managing director of the Power-Gas Corporation Ltd. and Ashmore, Benson, Pease & Co., of Stockton-on-Tees. He joined the first mentioned company in 1917 and was appointed to the board of directors in 1948. He is also a director of an associate company, Rose, Downs & Thompson Ltd., of Hull.

New hand at the helm of I.C.I.

Dr. Alexander Fleck will succeed Mr. John Rogers as Chairman of Imperial Chemical Industries, Ltd., on June 30. He will be the fourth chairman of I.C.I. Like his immediate two predecessors (Lord McGowan retired in December, 1950), Dr. Fleck was born in Glasgow. He is 65 and started with the company in 1917. He became works manager of the Widnes works and joined the main I.C.I. board in 1944 when he was made responsible for the Billingham factory. In 1946 he was put in charge of developing the nearby Wilton project and in December, 1950, at the time of Mr. Rogers' appointment to the chairmanship, he was made a deputy chairman. Dr. Fleck is a chemist.

I.C.I. board changes

Sir Wallace Akers and Mr. W. F. Lutyens retired from the board of Imperial Chemical Industries Ltd. on April 30. Sir Wallace's place as research director is taken by Dr. R. Holroyd. Mr. Lutyens is succeeded as development director by Mr. C. R. Prichard.

Sir Wallace Akers was with Brunner, Mond & Co. Ltd. from 1911 to 1924. In 1928 he joined I.C.I. Technical Department in London and in 1931 was appointed chairman of the delegate board of I.C.I. (Fertiliser & Synthetic Products) Ltd. In 1939 he was appointed an I.C.I. executive manager and was appointed to I.C.I. board in 1941 as the director responsible for research. During the last war Sir Wallace Akers was Director of Atomic Energy for the Ministry of Supply. He was knighted in 1946. As reported in our Editorial notes, he has just been appointed to the committee set up by the Government to devise a new organisation for atomic energy development.

Mr. W. F. Lutyens joined Brunner, Mond & Co. Ltd. in 1913. He was appointed chairman of the delegate board of I.C.I. (Alkali) Ltd. in 1931. In 1939 he was appointed an I.C.I. executive manager and was appointed to the board of I.C.I. in 1941 as the director in charge of development. Mr. Lutyens was I.C.I. development director from 1941 to 1948, when he was made responsible for the Heavy Chemicals Group. In 1951 he again took charge of development and was also made responsible for the *Terylene* Council.

Dr. Ronald Holroyd, a Yorkshireman by birth, was educated at Archbishop Holgate's Grammar School and Sheffield

Mr. R. W. Rutherford

University, where he did research in fuel technology and pure chemistry. During his service with I.C.I. he has been continuously connected with hydrogenation and oil research, mostly at the Billingham Division. He joined this Division's board in 1947, and was appointed joint managing director in 1951. He was appointed to the I.C.I. board in 1952.

Mr. C. R. Prichard was educated at Winchester and Trinity College, Oxford. He joined Brunner, Mond & Co. in 1926, and served with this company and I.C.I.'s Alkali Division—its successor—from this date until 1950, except for a period of war service with the R.A.F. In 1945 he became joint managing director of the Division, and five years later was made chairman of the Salt Division. He was appointed to the I.C.I. board in November 1952.

Oil group's £39 m. equipment order

Export orders totalling nearly £39 million, compared with just over £26 million in 1951 and representing some 2½% of the whole U.K.'s materials exports, were placed by the Royal Dutch Shell Group of oil companies last year. This sum was in addition to the expenditure incurred in this country both for the continuation of the refinery programme and for new tankers for the Shell fleet, already one of the world's largest maritime operators.

More than 450 British companies have been commissioned to supply a great variety of export articles ranging from huge refinery distillation columns, aircraft and specialised oilfield vehicles and drilling equipment to small office and domestic stores.

Among the manufacturers who received substantial orders, some running into several million pounds, were Richard Thomas Steel Co. of Wales Group for tinplate, etc., Stewarts & Lloyds Ltd. for steel pipes and Whessoe Ltd., mainly for storage tanks.

HUNGARY

Cellulose-from-straw factory

The production of cellulose from straw is to be started in Hungary in a new factory due for completion on September 17. As the country is poor in timber, cellulose has in the past had to be imported.

Previous attempts to make cellulose from straw produced a material suitable only for poor-quality brown paper. The new process, based on experience in the Soviet Union and East Germany, gives a high-quality white cellulose.

The new factory is at Szolnok, on the River Tisza, central Hungary. Bales of straw will enter the factory on conveyor belts and in a journey of 500 yds. through processing equipment and presses will be untouched by hand until it emerges as cellulose. Only 80 workers will be needed to operate the plant through the three shifts per day.

PORTUGAL

Fertiliser production to be increased

The new six-year plan for economic development includes an allocation of 165,000 contos for further expansion of existing ammonium sulphate plants.

Although superphosphate is produced in sufficient quantities, the government observes that there is considerable need for expanded output of nitrogenous materials.

The rate of consumption is increasing, but it is still far below that of other countries. Furthermore, the country must depend on foreign sources for adequate supplies, which is a drain on exchange reserves and may mean shortages in periods of international emergency.

It is hoped to increase annual output to 114,000 metric tons, which is estimated to be approximately sufficient for national requirements. This expansion is expected to be achieved in the first four years of the plan, but the success of the project is directly linked to the major programme of hydroelectric plant development, as the supply and cost of electric power is a principal factor.

NETHERLANDS

Cadmium plant planned

The Maastrichtse Zinkwit Maatschappij (Maastricht Zinc White Co.) has asked permission from the provincial authorities to build a new plant at Limmen (Limburg) for the production of cadmium. The company started making cadmium shortly before the war, but had to stop operations. Initial production will total about 50 tons annually.

SWEDEN

Zinc, lead and copper output to be increased

With the completion of new plant at the mines of A.B. Zinkgruvor in West Sweden, the mining capacity of this firm has now been increased from 100,000 tons to about 250,000 tons of ore annually. The ore, which is of good quality, yields zinc, lead, copper and a little gold and silver.

UNITED STATES

Synthetic rubber plants for sale

President Eisenhower has recommended the sale of 550 million dollars' worth of government-owned synthetic rubber plants to private industry. In a special message, the President asked Congress to enact legislation authorising disposal of the facilities.

'I am in hearty accord with the policy determination of the Congress that the security interests of the nation will best be served by the development within the United States of a free competitive synthetic rubber industry, and I believe that now is the time to undertake plant disposal,' the President said. 'The programme recommended in the report of the Reconstruction Finance Corporation appears to provide the basic outline of a satisfactory method to achieve this result.'

Oil refinery modernisation

The Amarillo, Texas, refinery of the Texas Co. is to be completely modernised at a cost of several million dollars. The programme will virtually double the current capacity of the refinery. New design units will include crude distillation, delayed coking, *Orthoflow* fluid catalytic cracking, and catalytic polymerisation. The first two of these are integrated into a combination unit to achieve lower initial investment cost and reduced operating expense.

Approximate capacities in barrels/stream day of feed are: crude distillation 15,500, delayed coking 3,600 and catalytic cracking 6,000 (about 10,000 with recycle). The polymerisation unit is rated at about 470 barrels stream day of polymer gasoline. Construction is scheduled to start early this summer, the contractors being the M. W. Kellogg Co.

'Imports threaten chemical industry'

The Synthetic Organic Chemical Manufacturers' Association has told the Ways and Means Committee of the House of Representatives that the U.S. chemical industry is exposed to 'serious threatened injury' through imports mainly from Germany, Britain, France, Italy and Switzerland. The Association complained that the industry had been placed at a great disadvantage through 'drastic' tariff concessions granted to foreign manufacturers whose production costs were much lower than those in the U.S.

Mr. Cary Wagner, president of the Association, said imports of coal-tar chemicals had more than doubled between 1950 and 1951. He said the 'trade not aid' idea could be ruinous to many American industries which were not able to use mass-production methods with their products.

Isocyanates to be made

The Monsanto Chemical Co. is planning to build a multi-million-dollar plant for the manufacture of isocyanates. Major factor in this decision is the steadily increasing number of marketable products in which isocyanates are an essential component. Promising uses are seen in the production of the new synthetic rubbers and isocyanate - based foamed - in - place plastics.

It will be recalled that the new 'super' rubber, *Vulcollan*, is made with the use of naphthalene di-isocyanate (CHEMICAL & PROCESS ENGINEERING, February 1953, p. 35).

U.S.-European licensing proposals

Thirty new licensing proposals have been announced by the Mutual Security Agency Mission to the U.K. This bring the total number of proposals to 307. Included in the latest proposals are:

The American Engineering Co., of Philadelphia, Pa., desires to be licensed by a European firm for the eventual manufacture of a fluid power pump. Design and general know-how for vane-type pumps capable of pumping oil at high pressure are desired (No. 278).

The Bartelt Engineering Co., Rockford, Illinois, wishes to license the European manufacture of their packaging machines for which patents have been applied in Europe (No. 280).

Chicago Blower Corporation, Franklin Park, Illinois, offers new lines and all know-how on axial and centrifugal fan and blower manufacture for heating, ventilating and air conditioning (No. 281).

Larsen Products Corporation, Washington, D.C., wish to license European manufacture or distribution of a new plaster product of liquid plastic emulsion (No. 289).

Lincoln Park Industries Inc., Lincoln Park, Michigan, desire to be supplied by a European firm with equipment and know-how for the manufacture of dial indicators and air gauges (No. 290).

The Vapor Chemical Co., Rives Junction, Michigan, offer patents, processes and technique with any necessary equipment to European firms willing to manufacture their electric vaporisers for insect destruction deodorising (No. 305).

JAPAN

Synthetic rubber plant planned

A group of 11 Japanese industrialists interested in establishing a synthetic rubber plant in Japan visited the United States recently to investigate technical and construction phases of the industry and to obtain information on construction of plant for manufacturing butadiene and styrene, and copolymerising. The group represents the newly organised Synthetic Rubber Manufacturing Project Committee in Tokyo.

The project reportedly envisages establishment of a synthetic rubber plant capable of producing 36,000 tons of rubber annually. The final product would be a compound of butadiene and styrene, the former to be produced from alcohol extracted from potatoes.

PAKISTAN

U.S. finances new fertiliser factory

The United States Government is to contribute a further $3 million in 1953 and $5 million in 1954 for the construction of a fertiliser factory in Pakistan. It has been estimated that the plant will cost Rs. 55 million and that the factory will go into production by 1955. The U.S. Government contributed $4 million for the same project in 1952.

CEYLON

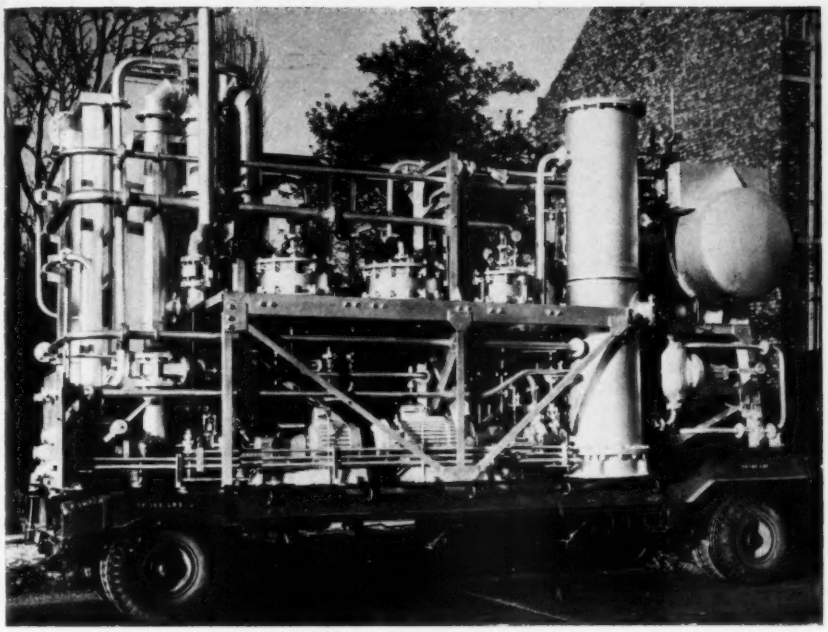
Glass factory closed down

The government glass factory closed down at the end of February. Its chief output consisted of tumblers and lamp chimneys. The factory was first set up during the last war to help overcome a general shortage of imported goods, but the local products have since been less and less able to compete with imported glassware. Efforts to dispose of the factory as a going concern have so far proved unsuccessful.

New decision on chemical projects

The Cabinet Economic Planning Committee has decided that, of the proposed industrial development projects set aside last October for further consideration following the publication of the World Bank Mission report, only three are to be proceeded with immediately. They are the paper mill, the vegetable oil factory and the DDT/caustic soda plant. In arriving at this conclusion, the committee has taken into account that Rs. 25 million out of an estimated total cost of Rs. 55 million has already been spent on these schemes, and that a considerable quantity of machinery ordered has already arrived in the island. Each project is expected to be floated as a public corporation with government participation.

The paper factory has been designed to produce about 4,000 tons p.a. of writing and printing paper from local paddy straw and illuk grass, plus enough kraft paper (from imported pulp) for the manufacture into paper bags for use by the Kankesanturai cement factory. The vegetable oil factory is expected to produce annually approximately 3,800 tons of low-grade oil by solvent extraction process from coconut oil cake, 800 tons of glycerin, 4,800 tons of fatty acid, 1,000 tons of lauryl alcohol and 50,000 tons of cattle food. The caustic soda/DDT plant will utilise solar-evaporated salt from adjacent salterns to produce, in the first instance, up to 5 tons of caustic soda per day. Provision has been made to incorporate a DDT- and chlorine-producing section, with an output of 2 tons and 1 ton day, respectively. A proportion of the chlorine will be used for the manufacture of bleaching powder. The remainder of the reserved projects, including the proposed government textile mill, have been placed in cold storage until such time as improved economic and financial conditions permit their further review.



'PACKAGE' CO_2 PLANT

One of a number of 'Package Units' for the production of carbon dioxide built by the Power-Gas Corporation in association with The Girdler Corporation of U.S.A. The process used involves the combustion of oil fuel followed by treatment of the products of combustion with an absorbing agent which is regenerated by the heat of the combustion.

The report of a commission set up last year to investigate government industrial undertakings is expected to be published soon. A major recommendation is that certain government-controlled ventures should be formed into public corporations, financed by State and private capital, and in addition that, in the case of large undertakings such as the local production of fertilisers, foreign concerns with specialised experience should be invited to participate.

INDIA

New oil refinery project

Construction of a refinery to handle 500,000 long tons p.a. of crude petroleum is expected to begin at Visakapatnam early in 1955. The site has already been selected and the refinery will be owned and operated by an Indian company to be formed by an American firm. The source of supply has not yet been decided and, if a source near Visakapatnam is decided upon, the annual capacity of the refinery may be stepped up.

This project is the result of an agreement recently signed between the American firm and the Indian Government. Two years ago a similar agreement was signed with a British and an American firm for setting up two oil refineries on Trombay Island, Bombay. When all three refineries go into full production, they will have a total capacity of about 3.8 million tons p.a. of crude oil. This will mean that India will be practically self-supporting in petroleum products and will be able to build up a chemical industry to utilise the by-products.

Plans for atomic power

Included in the Indian Government's seven-point plan for the development of atomic energy are the construction of an atomic reactor and the setting up of a pilot plant for the extraction of uranium from copper tailings and low-grade uranium ore. Other plants projected are for processing thorium and uranium from residual cake left after rare earth chlorides and carbonates have been extracted from monazite, and for processing uranium to the state of atomic purity.

Speaking on the peaceful uses of atomic energy in Bombay, Sir John Cockroft, director of the Atomic Energy Research Establishment at Harwell, said the coal resources of the world were going down and the situation would get worse as oil reserves were depleted. There was need for an additional source of power for general use, and nuclear power could meet this need when fully developed.

BRAZIL

French help for alkali projects

Contracts have been signed with a group of French banks for financing the project of Cia. Nacional de Alcalis for large-scale production of soda ash and caustic soda at Cabo Frio.

Foreign financing is reported to be equivalent to $12 million, to be spent on imported machinery and equipment. The National Bank for Economic Development undertakes to provide 180 million cruzeiros to meet local currency expenditures. The bank also will act as supervising agent for the Brazilian Government.

The proposed plant, which will use the *Solvay* process, is scheduled to produce eventually 100,000 metric tons of soda ash and 40,000 tons of caustic soda annually, as well as various by-products. Part of the soda ash will be used for causticising purposes.

Production for consuming industries would be substantially less than the total of 140,000 tons. Since annual requirements of alkalis are about 160,000 tons and the annual rate of increase is estimated at 15%, the factory, even when it reaches capacity, can meet only about 60% of domestic needs.

Imports of soda ash and caustic soda totalled approximately 110,000 tons in 1952, which, however, is not a representative period. Because of the scarcity of supplies on the market at the beginning of the year and the acute shortage of foreign exchange later, Brazil could not buy the quantities desired. Annual average imports of alkalis in 1950 and 1951 were 155,000 tons.

The plant has been studied for several years, and supporters of the project believe that it would save large amounts of foreign exchange. Some doubts have been expressed over the desirability of locating the plant at Cabo Frio because the necessary raw materials are not believed to be readily available in sufficient quantities. Electric power also will have to be provided in an area already lacking sufficient industrial power resources.

NORWAY

Big ore-handling plant

A new plant for handling iron ore at Narvik, north Norway, now under construction, will handle about 8,000 tons/hr. of ore. The plant, which will have a storage capacity of 4 million tons of iron ore, will be all-automatic. Four new ore crushers are being installed in a large chamber 70 ft. under the ground. For moving the ore, transport belts and cranes will be used, operating on a bridge 75-ft. high and almost 3,000-ft. long. Part of the installation will be ready next year and the remainder in 1955.

It is claimed to be the biggest plant of its kind in the world.

International atomic energy talks

Eleven countries have so far accepted invitations to attend an international conference on atomic energy from August 11 to 13. The first conference of its kind in Norway, it will be held at Kjeller, north of Oslo, where the Norwegian-Dutch atomic centre is now operating.

The nations that so far have indicated their intention to attend are Sweden, France, Argentina, Mexico, Switzerland, Germany, Jugoslavia, Italy, Spain, India and Belgium. In addition there will, of course, be delegates from Norway and its partner in atomic research, Holland. Among the subjects to be discussed is the

construction of heavy-water reactors for various purposes.

At the Norwegian-Dutch atomic institute at Kjeller 82 men and women are now employed, including 16 Norwegian research workers, 7 from Holland, 1 American, 1 Swiss and 1 Jugoslav. The rest of the staff are technicians and administrative workers.

The institute is operating in the present budget year on grants totalling £100,000. Of this amount a quarter is a direct grant from the Norwegian State and another quarter is an allocation from the profits of the State football pool. There is also a Dutch contribution of £17,000, which will be increased to £40,000 in the next budget year starting in July.

The institute, which has a reactor with heavy water supplied by Norway and uranium supplied by Holland, is now producing radioactive isotopes of various kinds. Radioactive iodine is being produced to fight thyroid cartilage disease, and preparations are in hand to produce radioactive phosphorus for use in counteracting various blood diseases, and radioactive gold for tumour injection. Radioactive cobalt, which is considered increasingly important in fighting cancer, is also to be made. For research in animal and plant biology, work is proceeding with the production of radioactive carbon.

Our Contemporaries—June

Manufacturing Chemist—Commercial Chromatography; Phosphoric Acid and Phosphates; Odour Research; Cost Control in the Chemical Industry; Progress Reports on Perfumery and Antibiotics.

Atomics—British Instruments for Atomic Technology; Airborne Radiation Detector; Atomics in Industry, 1.

Building Digest—Developments in Office Lighting; Roumanian Architecture Today.

Food Manufacture—Sharp's Toffee Factory, Maidstone; Transport and the Food Industry; Pressure Cooking and Cooling.

Muck Shifter—Essential Factors in the Design of Bulldozers and Scrapers; The Elements of Tidal Power.

Petroleum—Separation of Isometric Hydrocarbons; Government-sponsored Research in Britain, 1951-52; A New Use for Used Lubricating Oil; Oilfield Fossils of the Middle Eocene; Combined Firing in Steam Generators.

World Crops—The Influence of Introduced Crops on the Economy of Tropical Peoples; The Potentialities of Rubber as a Raw Material in Agricultural Machinery Construction; Aldrin and Dieldrin, Part 1; The Ring Culture Method.

Paint Manufacture—Wrinkle Finishes; Insulating Finishing Varnishes; A History of Paints and Varnishes in Great Britain.

MEETINGS

Incorporated Plant Engineers

June 9. 'Combustion heat and power,' by C. F. Roberts, 7.15 p.m., Engineers' Club, Albert Square, Manchester.

June 9. 'Thermal insulation of plant and buildings,' by C. G. Bower, 7 p.m., Royal Society of Arts, John Adam Street, Adelphi, Strand, London, W.C.2.

June 11. 'The plant engineer and the Factories Act,' by A. Hewitt, 7.30 p.m., Roadway House, Oxford Street, Newcastle-on-Tyne.

June 15. 'Industrial smoke abatement,' by P. H. Saunders, 7.15 p.m., Radiant House, Bold Street, Liverpool.

June 16. 'Dust extraction,' 7.30 p.m., Peahen Hotel, St. Albans.

International conferences

June 15-17. Eighth Nordic Chemistry Conference, Kjemisk Institutt Blindern, Oslo, Norway.

June 21-25. Annual Meeting of the American Society for Testing Materials, Atlantic City, N.J., U.S.A.

June 21-25. Thirteenth Annual Meeting of the Institute of Food Technologists, Boston, Mass., U.S.A.

June 22-26. Thirteenth International Dairy Congress, The Hague, Netherlands.

June 22-*July* 4. International Electrotechnical Commission Meetings, Opatija, Yugoslavia.

June 24-26. Sixteenth Congress of the Organisation for the Advancement of Spectrographic Methods, Paris.

June 29-*July* 3. Annual Congress of the South African Association for the Advancement of Science, Bulawayo, Southern Rhodesia.

CHEMICAL & PROCESS ENGINEERING

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We wish to know names and addresses of suppliers of the following:

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Recent publications

New resins. Just as the first synthetic resins exhibited great advantages over natural gums and resins, they in turn are now outstripped by the great advance presented in *Epikote* resins. These resins are condensation products of epichlorhydrin and diphenylolpropane. Their development is another instance of how the 'chemicals-from-petroleum' industry—in the growth of which Shell has played a leading part—is meeting the need for specialised chemicals of high quality, produced in abundance. In an attractive new booklet, Shell claim that formulations based on *Epikote* resins retain the four major properties—outstanding toughness, flexibility, adhesion and resistance to chemicals and solvents, combining them in a balance hitherto impossible to achieve.

Vibration suppressors. Various types of vibration insulators are described in a new leaflet from the British Tyre & Rubber Co. Ltd. These machinery mountings are made of rubber springs bonded to metal supports and used in shear to give spring deflections. The leaflet gives details of maximum static loading, deflection and dimensions of the various types. Vibro-insulators have many uses, for example in heavy switchboards, looms, fans, diesel engines, generators, refrigeration units, small lighting sets, etc. A guide to the selection of the correct type of B.T.R. vibro-insulator and the correct method of use is also included.

Driers and coolers. A new brochure describes the Sturtevant rotary drier, which has been found well suited to the rapid removal of moisture from large quantities of loose material such as coal, sand, slag, gypsum, fertiliser, etc. The driers are of the single-shell type, usually inclined but sometimes horizontal, and designed to ensure a rapid and continuous drying process. There is a section on the arrangement of these rotary driers in various types of installations. Sturtevant machines for degreasing, washing and drying small metal parts are also featured in the brochure, as are rotary coolers and auxiliary apparatus for use with the rotary driers.